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(71) Applicant: GORE ENTERPRISE HOLDINGS, INC. [US/US]; 551 Paper Mill Road, P.O. Box 9206, Newark, DE 19714 (US).

(72) Inventors: DOVE, Kevin, E.; 2605 Whittier Place, Wilmington, DE 19808 (US). EGRES, Ronald, G.; 12 Highland Circle, Newark, DE 19713 (US). MINOR, Raymond, B.; 233 Greenhaven Drive, Elkton, MD 21921 (US). MILLS, David, J.; 405 Nonantum Drive, Newark, DE 19711 (US). HISANO, Hirokazu; 10-2, Satsuki-cho, Ako-shi, Hyogo 678-0226 (JP). WENDL, Manfred; Heubergstrasse 18, D-83026 Rosenheim (DE).

(74) Agents: CAMPBELL, John, S. et al.; W.L. Gore & Associates, Inc., 551 Paper Mill Road, P.O. Box 9206, Newark, DE 19714-9206 (US).

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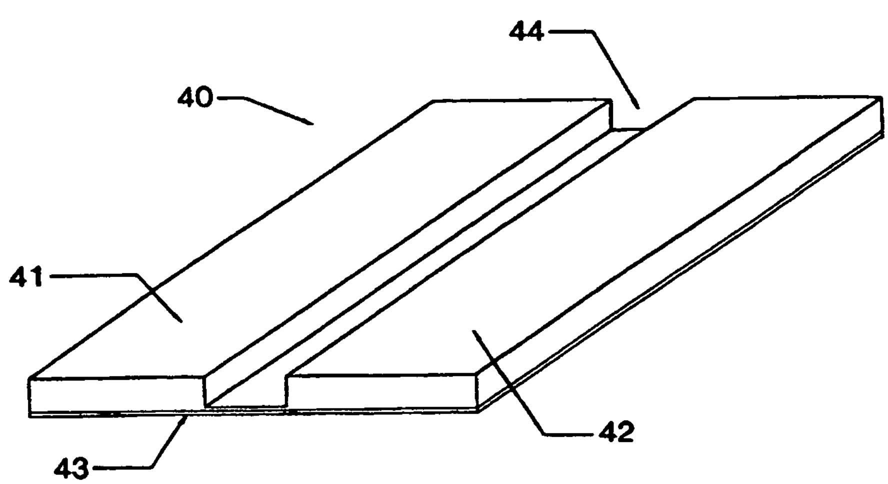
(54) Title: LOW STRESS TO SEAL FORM-IN-PLACE GASKET



(57) Abstract: A multilayer, unitary form-in-place gasket (40) including at least one inner layer of microporous expanded PTFE (421, 42) disposed between a first substantially air impermeable outer layer (43, 46) and a second substantially air impermeable outer layer (43, 46), and a substantially air impermeable region (43, 45) bridging the first and second substantially air impermeable layers (43, 46). The inventive gasket (40) forms a substantially air impermeable seal when compressed at low stress.

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TITLE OF THE INVENTION

LOW STRESS TO SEAL FORM-IN-PLACE GASKET

FIELD OF THE INVENTION

The present invention relates to gaskets and, more particularly, to a form-in-place gasket that forms a seal under less stress than required with existing gaskets.

BACKGROUND OF THE INVENTION

A wide variety of gaskets are known for use in sealing applications. Expanded polytetrafluoroethylene (PTFE) is widely used today as a gasket material. As disclosed in U.S. Patent No. 3,953,566 to Gore, this material has numerous properties making it highly desirable as a gasket. These properties include being readily compressible and conformable, being chemically resistant, having relatively high strength, and being far less prone to creep and loss of sealing pressure than non-expanded full density PTFE alone.

In many sealing applications, the gasket is used to seal the junction between flanges on equipment such as reaction vessels, storage vessels and flanged pipes. In such applications, expanded PTFE is a desirable material for the gaskets because the expanded PTFE gasket can be placed between the flanges, and the flanges can then be pressed together with the application of force, such as by tightening of bolts. This application of force compresses the expanded PTFE. As the expanded PTFE is compressed, its initial pore volume is reduced, thus densifying the expanded PTFE. Particularly with metal-to-metal flanges, it is possible to apply sufficient force (or "stress") to the flanges to fully densify the expanded PTFE. Thus, in at least part of the expanded PTFE gasket, the pore volume is reduced to substantially zero, such that a fluid contained within the pipes and vessels is prevented from leaking between the flanges by the densified, non-porous PTFE gasket, which seals the flanges.

In many applications, particularly when harsh chemicals are used which would readily break down the metal or the metal could contaminate the chemical which is being transported or housed, it is common to use steel

equipment that is lined with corrosion resistant material, especially glass or ceramic. Since this lined equipment is so often used with extremely harsh chemicals, there is great desire to use PTFE gaskets to seal the connecting flanges of this equipment because of the well-known extraordinary chemical resistance of PTFE. Unfortunately, non-expanded full density PTFE gaskets are generally not conformable enough to effectively seal this type of equipment. Particularly, in the case of glass-lined steel flanges, although there is a relatively smooth finish, there is often a large amount of unevenness or lack of flatness associated with the flanges. This unevenness or lack of flatness requires the gasket to have to conform to large variations around the perimeter as well as between the internal and external diameter of the flange in order to create an effective seal. Thus, a non-expanded full density PTFE gasket is not conformable enough to seal many of these applications.

Because expanded PTFE is so conformable, it would be desirable to use expanded PTFE to seal these commonly uneven flanges. Unfortunately, in many of these applications it is not possible to apply sufficient force to the flanges to create enough gasket stress to fully densify the expanded PTFE gasket to create an effective seal. For example, glass-lined steel flanges for vessels and pipes may deform, fracture, or break upon the application of a high amount of stress. Thus, in these applications, an expanded PTFE gasket may not be completely densified to reach a non-porous state, and therefore does not become leak proof, because the maximum stress that can be applied to the flanges without breaking them is not sufficient to so densify the gasket.

In many cases, it is not only necessary to be able to seal the actual fluid being housed or transported, but it is additionally necessary for the gasket to provide an air tight seal which can pass what is commonly known in the industry as a "bubble test". It is common to run this type of test as a pre-start-up qualifying test for checking for leaks in piping systems before allowing the system to be used in production carrying the actual fluid for which it was intended. In this test, the gasketed vessel and piping systems are pressurized with air and then sprayed with soapy water. The vessel and piping flange assemblies are visually checked for bubbles appearing in the soapy water indicating air leakage. All leakage sites must be eliminated to pass the bubble test.

Thus, what has been desired for many years is an easy-to-use highly chemically resistant gasket, which can effectively conform and provide an air

tight seal for this equipment with the low loads or stresses that are available to create the seal.

There have been many attempts to provide a gasket that can effectively seal these difficult applications. Most of these attempts involve a two-piece gasket. These gaskets are commonly referred to as envelope gaskets. In most envelope gaskets, an outer envelope of PTFE is formed and is then separately filled with a more compressible filler material such as compressed asbestos or other felted gasket filler, an elastomer or plastic material, or a corrugated ring of metal, usually stainless steel. The basic concept is the PTFE jackets for the envelope gaskets provide chemical resistance while conformability is provided by the filler material.

Unfortunately, as explained in US Patent No. 4,900,629 to Pitolaj, envelope gaskets are subject to a number of disadvantages. The envelope jacket often will fold over on itself during installation of the gasket, thereby creating creases in the gasket that cause leaks. Also, there may be pinhole leaks in the envelope itself, causing corrosive material to attack the envelope filler resulting in degradation of the filler. When the filler degrades, sealing stress can be diminished, causing a leak to occur. Another problem, which can result, is that the degraded filler material can contaminate the fluids that were contained within the pipe or vessel. In some instances, the envelope jacket of PTFE will separate from the conformable filler material and ripples or folds may occur merely from stretching the envelope over the filler, again causing leaks to occur. Also, if uneven flange torquing occurs, the jacket may become overstressed and burst, once again allowing the corrosive material to attack the filler resulting in degradation of the filler and loss of the seal. Another problem is that these envelope gaskets are also subject to cold flow or creep, which requires periodic bolt retorquing.

In US Patent No. 5,195,759 to Nicholson, an envelope gasket is employed with a PTFE envelope within which is an elaborate metal filling consisting of wound or nested turns of thin metal strips perforated to provide resilience in the direction of their width. Individual turns can move or collapse to different extents, thereby accommodating lack of flatness of the surfaces to be sealed. Turns of fluid-impervious material may be distributed among the turns of the perforated strips. Although the gasket has some advantages, it still suffers from many of the disadvantages mentioned above associated with envelope gaskets, such as chemical attack of the metal filling under certain conditions.

In US Patent No. 5,558,347 to Nicholson, a gasket is disclosed comprising an envelope of chemically resistant PTFE and a metallic packing ring within the envelope is shaped to form cells. The cells may be filled with an inert gas under pressure so that increased loads on the gasket may be cushioned. Although this gasket also has some advantages, it still suffers from many of the same disadvantages mentioned above associated with envelope gaskets.

In Japanese Laid–Open Patent Application Number 4-331876 to Ueda et al., another envelope (jacket) gasket is proposed in which the outer periphery of a core composed of low-density porous PTFE that has been fibrillated (expanded) and has a density of 1.8 g/cc or less is covered with a sheath composed of high-density sintered PTFE. Although this gasket has the benefit of being 100% PTFE, and therefore does not suffer the chemical attack problems resulting from pinhole leaks in the outer envelope, it can still suffer from the aforementioned problem of the outer envelope or jacket folding over on itself during installation of the gasket, thereby creating creases in the gasket that cause leaks. It can also suffer from the aforementioned problem of the envelope jacket of PTFE separating from the conformable filler material creating ripples or folds that can result in leaks. Another problem with this gasket is that there is not a tight fitting contact between the envelope jacket and the inner porous PTFE core along the inner diameter of the gasket, thus leaving the envelope jacket without a backing in this area, and therefore more susceptible to damage during installation and while in use.

As mentioned in US Patent No. 4,900,629 to Pitolaj, in an attempt to rectify some of the problems associated with envelope gaskets, a homogeneous PTFE gasketing material filled with microbubbles (i.e., glass microballoons) was developed. This material, as illustrated by Garlock Style 3504 gasketing manufactured by Garlock, Inc. of Palmyra, N.Y., uses glass microballoons to impart compressibility (25% to 35%) to a PTFE binder, thereby providing a more deformable gasket without the disadvantages experienced by multiple component gaskets. This homogeneous PTFE / microballoon gasketing material exhibits enhanced compressibility and sealing characteristics due to the incorporation of microballoons, while maintaining the resistance to chemicals and the enhanced temperature characteristics provided by PTFE. However, the addition of the microballoons to the PTFE lowers the tensile strength properties that would be provided by pure PTFE gasketing.

Plus, this gasket does not enjoy some of the aforementioned advantages that expanded PTFE has over non-expanded PTFE.

In US Patent No. 4,900,629 to Pitolaj, an attempt is made to overcome the inherent weakness of the homogeneous PTFE / microballoon gasket by loading more microballoons in the gasket surface layers, while leaving an unfilled PTFE center section. The microballoon filled layers are each formed to be within the range of from 20 – 25% of the overall thickness of the resultant gasket material, while the central PTFE section is within the range of from 50 – 60% of the overall gasket thickness. As explained in this patent, these ratios are important because if the outer surface layers are each formed to be below 20% of the overall gasket thickness, the finished composite sheet loses compressibility, while if they are formed to be above 25%, creep resistance and tensile strength are sacrificed in the finished product. Although this gasket is an improvement upon the homogeneously loaded microballoon gasket, and avoids the problems associated with envelope gaskets, it still does not adequately solve the problems of many applications. It is still left trying to trade off compressibility with creep resistance and tensile strength. This gasket also does not enjoy some of the aforementioned advantages of expanded PTFE compared to non-expanded PTFE.

In another attempt to rectify the two-piece nature problems associated with envelope gaskets, in US Patent No. 5,112,664 to Waterland, a unitary shielded gasket assembly is provided for use in corrosive environments having a synthetic rubber gasket as a core and a shielding material of expanded high density PTFE with an adhesive on at least one surface of the shielding material at least partially enveloping the surface of the core gasket. This gasket does not suffer from the wrinkles and folds that can result from a two-piece envelope gasket; however, it still suffers from the inherent problem of chemical attack problems resulting from pinhole leaks in the outer sheath.

In still yet another attempt to rectify the problems associated with envelope gaskets, in European Patent Application No. EP 0 736 710 A1, an annular gasket composed of porous PTFE for sanitary piping is proposed in which the surface layer of a gasket inner part directly contacting with sealed fluid is formed as a pore-free fused solidified layer. It is stated that the osmotic leak from the gasket inner part is prevented by the pore-free fused solidified layer formed in the gasket inner part although the gasket is composed of a porous material. Moreover, it is stated that since the fused solidified layer is formed only on the surface layer of the gasket inner part, the intrinsic

properties of porous PTFE such as flexibility and affinity are not spoiled. This gasket enjoys the benefits associated with a pure PTFE gasket; however, it can be difficult to attain a robust pore-free fused solidified layer that adequately resists permeation under stress. Furthermore, because of the rounded convex nature of the flanges of glass-lined steel, in many cases there is a ready leak path between the pore-free fused solidified layer formed in the gasket inner part of the gasket and where the flange contacts the gasket. This leak path is shown in Figure 20. This figure shows a side cross-sectional view of a gasketed flange assembly 90 of two conventional glass-lined steel flanges 96 which have the rounded convex mating edges 95 which contact the gasket 91 on part of its top and bottom surfaces 94. It can be seen that if only the surface layer of the internal diameter 93 of the gasket 91 is impermeable to the contained fluid, there is a ready leak path 92 through that exposed part of the gasket 91 which is not impermeable to the fluid.

It is often necessary to employ hoists and sometimes large cranes to remove and lift portions of glass-lined vessels in order to install a new gasket. Ancillary equipment such as agitators, baffles, and sensors utilized within the vessel make it extremely difficult, if not impossible, to install an envelope gasket or any other preformed gasket without significantly parting the vessel. Many times, the lid of a glass-lined steel vessel must be lifted a distance equal to the length of the equipment attached to it so that the gasket can be set in place. It would be desirable to provide a form-in-place gasket such that it does not require such a large distance of separation between the portions of the equipment in order to install the gasket.

It would be desirable to provide a unitary, conformable, creep resistant, high strength, chemically resistant, form-in-place gasket that can seal openings, especially glass-lined steel flanges, upon the application of a relatively low stress. It is therefore a purpose of the present invention to provide a unitary expanded PTFE form-in-place gasket that provides a substantially air impermeable seal only upon the application of a low stress.

SUMMARY OF THE INVENTION

The present invention provides a multilayer, unitary, form-in-place gasket including at least one inner layer of expanded PTFE disposed between

a first substantially air impermeable outer layer and a second substantially air impermeable outer layer, and a substantially air impermeable region bridging the first and second substantially air impermeable layers.

In another aspect, the present invention provides a multilayer, unitary, form-in-place gasket including a top surface, a bottom surface, an inside edge, and an outside edge; a first substantially air impermeable layer disposed on the top surface; a second substantially air impermeable layer disposed on the bottom surface; at least one layer of expanded PTFE disposed between the first and second substantially air impermeable layers; and a substantially air impermeable region bridging the first and second substantially air impermeable layers.

In another aspect, the present invention provides a multilayer, unitary, form in place gasket having a top surface, a bottom surface, an inside edge, and an outside edge, including a first chamber of expanded PTFE disposed adjacent to the inside edge having a first air impermeable top layer on the top surface and a first air impermeable bottom layer on the bottom surface; a second chamber of expanded PTFE disposed adjacent to the outside edge having a second air impermeable top layer on the top surface and a second air impermeable bottom layer on the bottom surface; and a substantially air impermeable region disposed between first and second chambers.

In still another aspect, the present invention provides a multilayer, unitary, form-in-place gasket having a top surface, a bottom surface, an inside edge, and an outside edge with a first chamber of expanded PTFE disposed adjacent to the inside edge having a first top portion on the top surface and a first bottom portion on the bottom surface, wherein the first top portion and the first bottom portion are less permeable to air than the expanded PTFE of the first chamber; a second chamber of expanded PTFE disposed adjacent to the outside edge having a second top portion on the top surface and a second bottom portion on the bottom surface, wherein the second top portion and the second bottom portion are less permeable to air than the expanded PTFE of the second chamber; and a region disposed between the first and second chambers, the region being less permeable to air than the expanded PTFE of the first and second chambers In alternative embodiments, the region may be disposed on either the inside edge or the outside edge.

In still another aspect, the invention provides a method of making a form-in-place gasket comprising the steps of:

(a) providing at least one layer of expanded PTFE;

(b) bonding a substantially air impermeable layer to the layer of expanded PTFE to form a multilayer, unitary composite;

(c) removing a portion of the layer of expanded PTFE over the air impermeable layer to form a channel; and

(d) folding the multilayer unitary composite around the channel.

DESCRIPTION OF THE DRAWINGS

The present invention is described herein with in conjunction with the accompanying drawing, in which:

Figure 1 is a perspective view of a gasket according to an exemplary embodiment of the present invention in an unfolded state;

Figure 2 is a perspective view of the gasket of Fig. 1 in a folded state;

Figure 3 is a perspective view of a gasket according to another exemplary embodiment of the present invention;

Figure 4 is a perspective view of a gasket according to another exemplary embodiment of the present invention;

Figure 5 is a perspective view of a gasket according to another exemplary embodiment of the present invention;

Figure 6 is a perspective view of a gasket according to another exemplary embodiment of the present invention;

Figure 7 is a perspective view of a gasket according to another exemplary embodiment of the present invention in an unfolded state;

Figure 8 is a perspective view of the gasket of Fig. 7 in a folded state;

Figure 9 is a perspective view of a gasket according to another exemplary embodiment of the present invention in an unfolded state;

Figure 10 is a perspective view of the gasket of Fig. 9 in a folded state;

Figure 11 is a perspective view of a gasket according to another exemplary embodiment of the present invention;

Figure 12 is a side cross-sectional view of the gasket of Figure 11;

Figure 13 is a perspective view of a gasket according to another exemplary embodiment of the present invention;

Figure 14 is a perspective view of a gasket according to another exemplary embodiment of the present invention;

Figure 15 is a top view of a form-in-place gasket according to the present invention being installed (formed) to seal a flange;

Figure 16 is a side cross-sectional view of a test fixture used to determine sealability of the exemplary embodiments of the present invention;

Figure 17 is a side cross-sectional view of a test apparatus used to measure air permeability on the exemplary embodiments of the present invention;

Figure 18 is a top view of a form-in-place gasket according to the present invention illustrating a method of joining the ends of the gasket to form a seal;

Figure 19 is a side cross-sectional view of a conventional prior art envelope gasket;

Figure 20 is a side cross-sectional view of two conventional glass-lined steel flanges with a prior art gasket between them;

Figure 21 is a perspective view of a test apparatus used to measure liquid permeability on the exemplary embodiments of the present invention;

Figure 21A is a side cross-sectional view of a gasket after being tested in the liquid permeability test apparatus of Figure 21;

Figure 22 is a perspective view of a form-in-place gasket according to the present invention illustrating a method of joining the ends of the gasket to form a seal;

Figure 22A is an exploded perspective view of a portion of the gasket of Figure 22 illustrating a method of joining the ends of the gasket to form a seal;

Figure 23 is a side cross-sectional view of two flanges with a form-in-place gasket according to the present invention between them;

Figure 24 is a graphical display of results from testing performed on the exemplary embodiments of the present invention;

Figure 25 is a perspective view of a form-in-place gasket according to the present invention illustrating a method of joining the ends of the gasket to form a seal;

Figure 26 is a perspective view of a form-in-place gasket according to the present invention illustrating a method of joining the ends of the gasket to form a seal; and

Figure 27 is a perspective view of a form-in-place gasket according to the present invention illustrating a method of joining the ends of the gasket to form a seal.

DETAILED DESCRIPTION OF THE INVENTION

The present invention provides an improved expanded PTFE form-in-place gasket that provides a substantially air impermeable seal upon the application of a relatively low load to the components joined or sealed by the gasket, thereby applying a relatively low stress to the gasket. By "air impermeable" as used herein is meant resistant to transport of air through a material. Permeability may be measured using any known technique. By "low stress" as used herein is meant a stress below that required to fully densify a porous expanded PTFE gasket (less than about 20,700 kPa (3000psi)). It generally takes at least about 20,700 kPa (3000 psi) to fully densify a porous expanded PTFE gasket. Most low stress applications generally apply less than about 10340 kPa (1500 psi) gasket stress, while some low stress applications may apply less than about 2070 kPa (300 psi) gasket stress.

By "form-in-place" is meant that the gasket must be reshaped or formed to the surface(s) to be sealed during the application of the gasket. Typical form-in-place gaskets are sold in a continuous flexible tape or cord form where the end user will cut off a suitable length of the tape or cord and then bend it into the shape of the surface to be sealed. For instance, in the case of a circular glass-lined steel flange of a vessel, the form-in-place gasket in the shape of a flexible tape will be bent into a circular shape such that the gasket shape will match the perimeter of the flange. Typically, an adhesive is used to hold the form-in-place gasket in this shape against the flange until the other mating flange is secured with the gasket compressed between the two flanges.

An exemplary embodiment of the present invention is shown in Figures 1 and 2. In this embodiment, gasket 40 comprises two sections, 41 and 42, of microporous expanded PTFE material with a layer of substantially air impermeable material 43 on one surface thereof. Between sections 41 and 42 is a cutout section 44. Cutout section 44 can range from being a very shallow cutout section (removing a small portion of a microporous expanded PTFE layer or layers that was originally present) or a very deep cutout section (removing almost all if not all of the microporous expanded PTFE layer or layers originally present).

In use, gasket 40 is folded around cutout 44 to form the configuration shown in Figure 2. In this present final configuration, gasket 40 has a microporous expanded PTFE layer 49, made of layers 41 and 42, covered on

three sides by substantially air impermeable layer 43. Substantially air impermeable layer 43 can be alternatively described as comprising top and bottom substantially impermeable layers 46a and 46b bridged by substantially impermeable region 45.

A variant of this embodiment is shown in Figure 3, where gasket 50 is substantially the same as the embodiment as shown in Figures 1 and 2, but does not include the cut-out portion 44.

Figure 15 illustrates the application of the form-in-place gasket 40 of Figure 2 to a flange 48. The flexible tape form of the gasket 40 is shown being reshaped or formed by hand to the shape of the flange 48 during the installation. The remaining ends 47a and 47b of the form-in-place gasket can be joined and sealed creating a sealed joint by using a variety of techniques. One such technique is to simply overlap the two ends 47a and 47b. The high level of conformability of the microporous expanded PTFE layer 49 of the form-in-place gasket 40 can accommodate this overlapped portion creating a seal at the overlapped joint. It is preferable to have the overlapped joint 55 at one of the bolt locations 56 of the flange 58, such as illustrated in Figure 18 to enable maximum gasket stress at this joint 55 of the form-in-place gasket 40 . A preferred variation of the overlap joint 55, which is illustrated in Figures 22 and 22A, includes producing a skive cut 59 at each end of the gasket 40 so that the extra thickness encountered at the overlapped joint is gradual. In the case of the embodiments such as shown in Figures 1-3 and 7-10 where the gasket is folded over in actual use, it is generally preferred to skive cut the ends in the unfolded configuration as shown in Figure 25 leaving the substantially air impermeable layer 43 as the long dimension of the skive cut 59, then folding the gasket over for the installation as shown in Figure 26. Figure 27 shows how two folded over skived ends 57a and 57b such as shown in Figure 26 can be overlapped to create the preferred joint 55. Thus, in this preferred joint 55 there is minimal, if any exposed microporous expanded PTFE 49 at the tips 54a and 54b of the ends 57a and 57b of the formed overlapped joint 55 that must be compressed to seal the joint 55.

Still other variations of joining the ends may include stripping some portion (or all) of the porous expanded PTFE from one end and tucking the other end of the gasket inside of the vacated space.

Another benefit of the embodiments where the gasket is folded over during installation such as shown in Figure 3, is that for large variations in the gap to be sealed between the flanges, it is easy to add a length of material

such as expanded PTFE between layers 41 and 42 of the folded configuration to serve as a shim, taking up the extra space of the large gap sections. It is generally preferable to skive cut the ends of the shim to better match the typically gradually increasing and decreasing nature of the extra gap section. The shim can be inserted very easily from the outer edge 51 of the gasket, while the substantially air impermeable region 45 at the fold serves as the inner edge 52.

Another exemplary embodiment of the invention is shown in Figure 4 wherein a gasket 60 is provided where a microporous expanded PTFE layer 61 is wrapped on four sides by a substantially air impermeable layer 62. Substantially air impermeable layer 62 can be alternatively described as comprising top and bottom substantially impermeable layers 66a and 66b bridged by substantially impermeable regions 65a and 65b. This embodiment is formed to the sealing surface in its present state, without the folding over step of the embodiments illustrated in Figures1-3.

Figures 5 and 6 illustrate other exemplary embodiments in which gasket 70 is formed of a microporous expanded PTFE layer 71 bounded on three sides by a substantially air impermeable layer 72. As shown in Figure 6, layer 72 may extend only part of the way across the surfaces of microporous expanded PTFE layer 71. Substantially air impermeable layer 72 can be alternatively described as comprising top and bottom substantially impermeable layers 76a and 76b bridged by substantially impermeable region 75. In these embodiments, the form-in-place gasket is not folded over prior to being formed to the sealing surface.

In use, for example gasket 40 of Figure 2, gasket 40 is subjected to the application of stress by the sealing surfaces, such as mating flanges, on the top and bottom substantially air impermeable layers 46a and 46b. Upon application of this stress, microporous expanded PTFE material of sections 41 and 42 compress somewhat, thereby reducing the porosity of expanded PTFE. Substantially air impermeable layers 46a and 46b are preferably thin such that gasket 40 can conform to any irregularities in the surface of the flanges to which they mate. This conformability is characteristic of the microporous expanded PTFE layers of sections 41 and 42 used with the gasket 40. Top and bottom substantially air impermeable layers 46a and 46b along with air impermeable region 45 serve to form an air impermeable barrier against the transfer of fluid from inside the vessel or pipes to the surface of the flanges where they may leak around gasket 40.

Additionally, in order to provide a form-in-place gasket that easily bends along the required circumference or other end use shape, it is important to minimize the thickness of the substantially air impermeable layer 43, especially that portion which forms top and bottom substantially air impermeable layers 46a and 46b. On the other hand, it is important to not be too thin, because then the air impermeable quality of the substantially air impermeable layer can be diminished. Thus, there is a balance of conformability and ease of bending versus impermeability that must be considered for the final application. Generally, the thicker the substantially air impermeable layer 43, the more impermeable the layer is. The thinner the substantially air impermeable layer 43, the less the conformability and ease of bending of the gasket 40 is affected. For some large diameter vessel flanges a substantially air impermeable layer of 1.0 mm thick or more may have the required ease of bending and conformability. However, for most of these applications, thicknesses equal to or less than 0.5 mm are generally even more useful, with thicknesses equal to or less than 0.15 mm generally preferred. In most applications where a relatively high level of conformability and ease of bending is desired, thicknesses equal to or less than 0.1 mm, 0.05mm and even 0.025 mm would be most preferred.

In one embodiment of the present invention illustrated in Figures 7 and 8, gasket 30 is formed in a similar manner to gasket 40 of Figures 1 and 2 except that the center portion of the microporous expanded PTFE section remaining from cutout section 34 is compressed to full density. This provides an additional layer or thickness of substantially air impermeable material 37 in addition to the substantially air impermeable material 33 in the region between microporous expanded PTFE sections 31 and 32. Thus, once the gasket is folded to the configuration shown in Figure 8, the two substantially air impermeable materials 37 and 33 combine to form a substantially air impermeable region 35 which is thicker than top and bottom substantially air impermeable layers 36a and 36b. One benefit of this embodiment is that the additional layer or thickness forming substantially air impermeable region 35 adds additional air impermeability to gasket 30 in a critical area of the gasket 30 which, in the folded over configuration of Figure 8 while in use, is oriented substantially perpendicular to the fluid to be sealed within a flange. This orientation is illustrated in Figure 23 with the arrow representing the direction in which the fluid to be sealed within the flange 39 strikes the gasket 30 substantially perpendicular to substantially air impermeable region 35. This

extra layer or thickness confined to this substantially perpendicular oriented region in this folded over configuration, however, does not significantly diminish the conformability or ease of bending of the gasket 30 to the necessary form-in place configuration required by the end use application. Additional layers or thickness added to top and bottom substantially air impermeable layers 36a and 36b can diminish conformability and hamper the ease of bending of the gasket. Thus, in this embodiment, the form-in-place gasket 30 derives extra impermeability in the critical substantially air impermeable region 35, without significantly diminishing conformability or ease of bending.

In another exemplary embodiment of the invention illustrated in Figures 9 and 10, a form-in-place gasket 110 is shown which is similar to the embodiment illustrated in Figures 7 and 8 except there is no cutout section. The groove 114 is produced entirely by compressing the microporous expanded PTFE layers in this region. Thus, the additional thickness substantially air impermeable region 115 in the folded over state of Figure 10 is made from combining compressed region 117 and that part of air impermeable layer 113 between sections 111 and 112 of the unfolded state of Figure 9. Thus, again in this embodiment, the form in place gasket 110 derives extra impermeability in the critical substantially air impermeable region 115, while maintaining thinner top and bottom substantially air impermeable layers 116a and 116b, therefore, not significantly diminishing conformability or ease of bending.

The microporous expanded PTFE layer of the embodiments (for example, microporous expanded PTFE sections 41 and 42 of Figures 1 and 2) can be made of a single layer of microporous expanded PTFE or of a plurality of individual layers of microporous expanded PTFE produced in a manner such as taught in U.S. Patent Nos. 3,953,566 and 4,187,390 to Gore.

The substantially air impermeable layers of the embodiments (for example, substantially air impermeable layers 46a and 46b of Figure 2) are preferably comprised of densified expanded PTFE. The densified expanded PTFE can be made by compressing the above described microporous expanded PTFE such as between two rollers. Densified expanded PTFE is preferred in that being PTFE it has the highest level of chemical resistance, while the expansion characteristics provide high levels of strength and creep resistance. Substantially air impermeable layers may in fact comprise a plurality of such densified expanded PTFE layers. Other substantially air impermeable materials may also be used, including

tetrafluoroethylene/hexafluoropropylene copolymer (FEP), tetrafluoroethylene/(perfluoroalkyl) vinyl ether copolymer (PFA), and skived PTFE. Alternatively air impermeable layers may be made of expanded PTFE impregnated with a filler such as an elastomer, a fluoroelastomer, a perfluoroelastomer, or a perfluoropolyether silicone elastomer. In general, the more chemically resistant the type of elastomer used or other type of nonpermeable coating or filler used, the more applications the gasket will be able to provide an effective sealing solution.

Substantially air impermeable regions of the embodiments (for example, substantially air impermeable region 45 of Figure 2) are preferably densified expanded PTFE although it may comprise any substantially air impermeable material, such a FEP, PFA and skived PTFE. Alternatively, substantially air impermeable regions may be made of expanded PTFE impregnated with a filler such as an elastomer, a fluoroelastomer, a perfluoroelastomer, a perfluoropolyether silicone elastomer, or any other type of nonpermeable coating or filler.

There may be applications where it is desirable to use different materials to form the substantially air impermeable layers versus the substantially air impermeable regions. Furthermore, there may be applications where it is desirable to use different materials to form one substantially air impermeable layer versus another or one substantially air impermeable region versus another. Additionally, there may be applications where it is desirable to use more than one type of material within a substantially air impermeable layer or a substantially air impermeable region. One such preferred combination of materials to produce a substantially air impermeable layer and / or region is a PFA / full density expanded PTFE composite. The full density expanded PTFE side is preferred to form the exposed outside surface of the substantially air impermeable layer while the PFA side is preferred to form the inside surface of the substantially air impermeable layer contacting the microporous expanded PTFE layer. In this type of an arrangement, the PFA portion of the composite provides extremely high levels of air impermeability for a relatively thin layer of material while the full density expanded PTFE portion of the composite provides extremely high levels of chemical resistance.

Another preferred combination of materials to produce a substantially air impermeable layer and / or region is an FEP / full density expanded PTFE composite for the same reasons as stated above.

In the exemplary embodiment illustrated in Figure 13 gasket 160 is formed of a microporous expanded PTFE layer 161 bounded on all four sides by an elastomer / expanded PTFE composite layer 163 serving as both the top and bottom substantially air impermeable layers 166a and 166b as well as forming the substantially air impermeable regions 165a and 165b on both the inside edge and outside edge.

Figures 11 and 12 illustrate yet another alternative embodiment of the present invention. In this embodiment, gasket 140 comprises two chambers or sections, 141 and 142, of microporous expanded PTFE material with a first top and bottom layers of substantially air impermeable material 146a and 146b on both top and bottom surfaces of section 141 and second top and bottom layers of substantially air impermeable material 146c and 146d on both top and bottom surfaces of section 142 thereof. Between sections 141 and 142 is a substantially air impermeable region 145 which bridges top and bottom substantially air impermeable layers 146a, 146b, 146c and 146d. Substantially air impermeable region 145 can be produced by compressing to full density the microporous expanded PTFE of that portion of the gasket 140 between the top and bottom substantially air impermeable layers 146a, 146b, 146c and 146d. Because the gasket embodiments of the present invention are intended for use in applications where there is low available load or stress, the microporous expanded PTFE layer(s) of the embodiments generally do not fully compress. Therefore, there is generally some porosity left in the microporous expanded PTFE layer(s) during use. In this embodiment, in use, gasket 140 is formed such that edge 148 of section 141 is formed to be the inside edge contacting the fluid to be sealed. It is thus possible for fluid contained within the sealed vessel or pipe to permeate through the microporous expanded PTFE material of section 141 in the direction of the arrow shown in Figure 12.

Substantially air impermeable region 145 prevents the escape of this fluid to the environment, however. Specifically, the fluid may permeate expanded PTFE material in first chamber 141 but is blocked from permeating into second chamber 142 by the substantially air impermeable region 145. In this manner, a leak-proof seal is provided.

It should be recognized that substantially air impermeable layers and substantially air impermeable regions of the embodiments would be substantially impermeable to fluids in general, including liquids, even low surface tension liquids, such as many solvents.

A desired advantage of the present invention in this embodiment shown in Figures 11 and 12, is that upon migration of fluid into the microporous expanded PTFE material of first chamber 141, and upon subsequent blockage of further fluid permeation by substantially air impermeable region 145, the fluid that is "trapped" in first chamber 141 exerts an outward force against first top and bottom substantially air impermeable layers 146a and 146b bordering section 141. This phenomenon helps further conform and seal first top and bottom substantially air impermeable layers 146a and 146b to the surfaces of the flanges, thereby improving the seal by gasket 140. Without being limited by theory, it is believed that second chamber 142 helps to provide a resistant force behind substantially air impermeable region 145 that helps prevent rupture of substantially air impermeable region 145.

The embodiment above can be advantageous in many applications where it is acceptable to have ingress of fluid into the gasket. There are some applications, however, where it is undesirable to have any ingress of fluid into the gasket, such as with many pharmaceutical applications. In these types of applications it is preferable to use the embodiments of the present invention which has an air impermeable region on the inner edge that prevents the ingress of fluid into the gasket.

In the exemplary embodiment illustrated in Figure 14 gasket 150 is similar to the embodiment shown in Figure 13 except the microporous expanded PTFE layer 151 is bounded only on one side edge and extending only partially across the top and bottom surfaces. Thus, the top and bottom air impermeable layers 156a and 156b and the air impermeable region 155 are formed from the elastomer / expanded PTFE composite. In application, this gasket may be applied by forming the gasket such that air impermeable region 155 is either facing the inside diameter (directly contacting the process fluid to be sealed) or bent in the opposite direction such that microporous expanded PTFE edge 158 is facing the inside diameter. In the applications where it is desired to prevent ingress of fluids into the gasket, it is preferred to form the gasket with substantially air impermeable region 155 directly contacting the process fluid. In applications where it is acceptable to have ingress of fluid into the gasket, it may be desirable to form the gasket such that microporous expanded PTFE edge 158 is directly contacting the process fluid. In this configuration, gasket 150 can enjoy the same type of sealing advantage explained above where the pressure of the process fluid could be used to help further conform and seal top and bottom substantially air impermeable layers

156a and 156b to the surfaces of the flanges, thereby improving the seal by gasket 150.

Another benefit of the embodiment shown in Figure 14 when formed in the configuration with microporous expanded PTFE edge 158 directly contacting the process fluid, is that the elastomer / expanded PTFE composite layer 153 can be protected in situations where the fluid to be sealed is a high surface tension fluid which has the capability to harm the elastomer / ePTFE composite layer 153, such as in the case with many acids. In the case of high surface tension fluids, the microporous expanded PTFE is already impermeable to the fluid, however, top and bottom substantially air impermeable layers 156a and 156b and substantially air impermeable region 155 are still needed in order to pass the required "bubble test" as previously explained. Thus, the elastomer / ePTFE composite portion 153 can provide the needed air impermeable nature of the gasket 150, while not being exposed to the contained fluid, such as with many acids.

To use as an example, gasket 40 of Figures 1 and 2, gasket 40 is preferably made by wrapping one or more layers of densified expanded PTFE on a mandrel to form an air impermeable layer 43; then wrapping one or more (preferably considerably more) layers of microporous expanded PTFE around the air impermeable layer 43 to form the microporous expanded PTFE material of sections 41 and 42. For both the densified expanded PTFE and the microporous expanded PTFE, it is most preferable to use PTFE which has been expanded in both longitudinal and transverse directions. As is taught in U.S. Patent No. 3,953,566 to Gore, the expansion of PTFE significantly increases its strength in the direction of expansion. By layering PTFE materials oriented in both the longitudinal and transverse direction, increased strength and resistance to creep and creep relaxation is imparted to the gasketing material. It is generally preferable to use unsintered densified expanded PTFE layers as opposed to sintered densified expanded PTFE layers to wrap on the mandrel to form air impermeable layer 43 to get a better bond to the microporous expanded PTFE material of sections 41 and 42. By "sintered" it is meant that the expanded PTFE has been exposed to temperatures in excess of 327°C, thereby creating an amorphous locking process as taught in U.S. Patent No. 3,953,566, and therefore reducing the crystalline content of the expanded PTFE. Additionally, a fluoropolymer such as FEP or PFA can be used to achieve a better bond between the densified expanded PTFE layer and the microporous expanded PTFE layer.

After heating the wrapped tube / mandrel assembly preferably above sintering temperatures (greater than 327°C and even more preferably greater than 345°C) to fuse the different layers into a unitary body, the wrapped tube may then be cooled and then spirally cut and laid flat or taken up in the form of a continuous tape. In this embodiment, two longitudinal slits are made in the microporous side of the tape at some depth into the microporous expand PTFE layer(s) taking care not to slit into the substantially air impermeable layer 43. The center section created by slits is then peeled from the tape producing a cutout section 44 of these layers of microporous expanded PTFE. In use, gasket 40 is folded around cutout 44 to form the configuration shown in Figure 2.

In the case of the embodiment shown in Figures 7 and 8, the center portion of the microporous expanded PTFE section remaining from cutout section 34 is subjected to a compressive treatment between, for example, two metal rollers, in order to compress this portion to full density, thus providing the additional layer or thickness of substantially air impermeable material 37 to the substantially air impermeable material 33 in the region between microporous expanded PTFE sections 31 and 32. Thus, once the gasket is folded to the configuration shown in Figure 8, the two substantially air impermeable materials 37 and 33 combine to form a substantially air impermeable region 35 which is thicker than top and bottom substantially air impermeable layers 36a and 36b. It may be desired to use heated rollers to aid in the compression step mentioned above.

It should be recognized that in certain applications it might be beneficial to have more than one substantially air impermeable region such that more chambers are created. These additional impermeable regions can be from combinations of the aforementioned embodiments from Figures 1-10, 13 and 14 or they can be from more than one air impermeable region contained between the inner and outer edges, such as the type shown in Figures 11 and 12. They may even include an impermeable region on the inner and / or outer edge with more than one impermeable region between the inner and outer edges. Thus, depending on the number and location of the air impermeable regions there may be more than two chambers within the gasket. One benefit of the multiple chambers is that the closed portions of the gasket could provide for an air cushioning effect in that increased loads on the gasket may be cushioned. Another benefit of having more than one air impermeable region is

that there are more air impermeable regions which must be traversed in order to create a leak path through the gasket.

It should also be appreciated that an additional distinct advantage of embodiments of the present invention over conventional envelope gaskets is the tight contact produced between the microporous expanded PTFE layer(s) to both the substantially impermeable layers and the substantially air impermeable region(s). This tight contact prevents the aforementioned problems associated with envelope gaskets pertaining to creating wrinkles, folds and creases in the jacket, which can cause leaks. The tight contact also provides backing to the substantially air impermeable region which makes it less susceptible to damage during installation and while in use. In Figure 19, a gasket is depicted which represents a typical envelope gasket, and in particular represents the jacketed gasket 80 disclosed in previously mentioned Japanese Laid–Open Patent Application Number 4-331876 to Ueda et al. This gasket 80 has free space 81 between the jacket or sheath 82 and the core 83. This free space (lacking tight contact) can be detrimental to the gasket in application due to the above stated reasons.

EXAMPLES

The present invention will now be described in conjunction with the following examples that are intended to illustrate the invention not to limit it. In the examples, the following test methods were used.

Example 1

A form-in-place gasket of the present invention was produced in the following manner. A continuous expanded PTFE sheet produced from fine powder PTFE resin through paste-forming techniques was obtained and expanded in directions 90 degrees opposed to each other (longitudinally and transversely) to form a microporous expanded PTFE sheet as taught in US Patent Nos. 3,953,566 and 4,187,390 to Gore. This sheet, having a thickness of about 0.015 mm was then rolled between two rollers at a fixed gap to compress the microporous expanded PTFE sheet into a full density non-porous expanded PTFE sheet. This non-porous full density expanded PTFE sheet had a final thickness of about 0.005 mm and a final width of about 1270 mm. One layer of this full density sheet was wrapped around a 584 mm diameter mandrel.

A roll of 0.013 mm thick PFA film commercially available from E.I. du Pont de Nemours, Inc., of Wilmington Delaware, under part number 50LP high performance PFA film was obtained. One layer of this PFA film having a thickness of 0.013 mm and width of 1524 mm was wrapped on top of the full density PTFE sheet on the mandrel.

A second continuous expanded PTFE sheet produced from fine powder PTFE resin through paste-forming techniques was obtained and expanded in directions 90 degrees opposed to each other (longitudinally and transversely) to form a microporous expanded PTFE sheet as taught in US Patent Nos. 3,953,566 and 4,187,390 to Gore. One hundred forty layers of this second microporous expanded PTFE sheet, measuring approximately 1600 mm wide and 0.038 mm thick was then wrapped on the mandrel covering the previously wrapped full density expanded PTFE sheet.

The microporous expanded PTFE layers were then secured at the ends of the mandrel to resist the tendency of this material to shrink back on itself at elevated temperatures. All the layers were then sintered while secured to the mandrel in an oven at 370°C for approximately 45 minutes to bond the layers together. After cooling, the bonded material was spirally cut from the mandrel in the form of a continuous tape having a width of approximately 125 mm. The final thickness of the continuous tape was 4.8 mm. The final thickness of the full density expanded PTFE / PFA composite film serving as the substantially air impermeable layer was 0.018 mm.

Example 1A

A 762 mm length of the tape from Example 1 was further slit into sections of width of 29 mm. Layers of the microporous expanded PTFE sheet were peeled from the tape to yield a tape having a total thickness of 1.6 mm. A channel 44 such as shown in Figure 1 was formed in the tape by making parallel cuts to the centerline of the length of the tape at a distance of 1.5 mm from the center. The depth of the cut was approximately 1.1 mm from the top of the tape where the PFA and full density PTFE films comprise the bottom of the tape. The microporous expanded PTFE material between the parallel cuts was peeled away and removed.

The tape was then folded in half as shown in Figure 2 resulting in a form-in-place gasket with a width of approximately 13 mm and a thickness of approximately 3.2 mm.

Example 1B

A section of length was cut from the continuous tape from Example 1. Layers of the microporous expanded PTFE sheet were peeled from the tape to yield a tape having a total thickness of 3.1 mm.

Example 1C

A 2134 mm length was cut from the continuous tape produced in Example 1. Layers of the microporous expanded PTFE sheet were peeled from the cut length of tape to yield a tape having a total thickness of 3.2 mm. This peeled down tape was then slit to produce a tape having a width of 86 mm. A channel 44 such as shown in Figure 1 was formed in the 86 mm wide tape by making parallel cuts to the centerline of the length of the tape at a distance of 3.2 mm from the center. The depth of the cut was approximately 2.2 mm from the top of the tape where the PFA and full density PTFE films comprise the bottom of the tape. The microporous expanded PTFE material between the parallel cuts was peeled away and removed.

The tape was then folded in half as shown in Figure 2 resulting in a form-in-place gasket with a width of approximately 40 mm and a thickness of approximately 6.4 mm.

Comparative Example 2

A sheet of 0.125 inch (3.2 mm) thick GORE-TEX GR® sheet gasketing commercially available from W.L. Gore & Associates, Inc. of Newark, Delaware, was obtained. This is a sheet of 100% microporous expanded PTFE.

Comparative Example 2A

An annular ring gasket was cut from the microporous expanded sheet material of Comparative Example 2. The annular gasket had an inner diameter of 152.4 mm and an outer diameter of 177.8 mm and was 3.0 mm thick.

Comparative Example 2B

An annular ring gasket was cut from the microporous expanded sheet material of Comparative Example 2. The annular gasket had an inner diameter of 610 mm and an outer diameter of 686 mm and was 3.0 mm thick.

Example 3

A form-in-place gasket of the present invention was produced in the following manner. A continuous expanded PTFE sheet produced from fine powder PTFE resin through paste-forming techniques was obtained and expanded in directions 90 degrees opposed to each other (longitudinally and transversely) to form a microporous expanded PTFE sheet as taught in US Patent Nos. 3,953,566 and 4,187,390 to Gore. This sheet, having a thickness of about 0.05 mm was then rolled between two rollers at a fixed gap to compress the microporous expanded PTFE sheet into a full density non-porous expanded PTFE sheet. This non-porous sheet had a final thickness of about 0.015 mm and a final width of about 1524 mm. Two layers of this full density sheet were wrapped around a 584 mm diameter mandrel.

One hundred forty layers of the second microporous expanded PTFE sheet from Example 1, measuring approximately 1600 mm wide and 0.038 mm thick, was then wrapped on the mandrel covering the previously wrapped full density expanded PTFE sheet.

The microporous expanded PTFE layers were then secured at the ends of the mandrel to resist the tendency of this material to shrink back on itself at elevated temperatures. All the layers were then sintered while secured to the mandrel in an oven at 370°C for approximately 45 minutes to bond the layers together. After cooling, the material was spirally cut from the mandrel in the form of a continuous tape having a width of approximately 125 mm. The final thickness of the continuous tape was 4.8 mm. The final thickness of the full density expanded PTFE serving as the substantially air impermeable layer was 0.030 mm.

Example 3A

A 762 mm length of the tape from Example 3 was cut into sections of width of 29 mm. Layers of the microporous expanded PTFE sheet were removed from the tape to yield a tape having a total thickness of 1.6 mm. A channel 44 such as shown in Figure 1 was formed in the tape by making parallel cuts to the centerline of the length of the tape at a distance of 1.5 mm from the center. The depth of the cut was approximately 1.1 mm from the top of the tape where the full density PTFE film comprises the bottom of the tape. The microporous expanded PTFE material between the parallel cuts was peeled away and removed.

The tape was then folded in half as shown in Figure 2 resulting in a form-in-place gasket with a width of approximately 13 mm and a thickness of approximately 3.2 mm.

Example 3B

A section of length was cut from the continuous tape from Example 3. Layers of the microporous expanded PTFE sheet were peeled from the tape to yield a tape having a total thickness of 3.1 mm.

Example 4

A form-in-place gasket of the present invention was produced in the following manner. One layer of the full density nonporous expanded PTFE sheet from Example 3, having a thickness of about 0.015 mm and a width of about 1524 mm, was wrapped around a 584 mm diameter mandrel.

One hundred layers of the second microporous expanded PTFE sheet from Example 1, measuring approximately 1600 mm wide and 0.038 mm thick, was then wrapped on the mandrel covering the previously wrapped full density expanded PTFE sheet.

The microporous expanded PTFE layers were then secured at the ends of the mandrel to resist the tendency of this material to shrink back on itself at elevated temperatures. All the layers were then sintered while secured to the mandrel in an oven at 370°C for approximately 45 minutes to bond the layers together. After cooling, the material was longitudinally cut from the mandrel in the form of a sheet that could be further cut into strips of tape. The final thickness of the sheet was 3.0 mm. The final thickness of the full density expanded PTFE serving as the substantially air impermeable layer was 0.015 mm.

Example 5

A form-in-place gasket of the present invention was produced in the following manner. Five layers of the full density nonporous expanded PTFE sheet from Example 1, having a final thickness of about 0.005 mm and a final width of about 1270 mm, were wrapped around a 584 mm diameter mandrel.

One hundred forty layers of the second microporous expanded PTFE sheet from Example 1, measuring approximately 1600 mm wide and 0.038 mm thick, was then wrapped on the mandrel covering the previously wrapped full density expanded PTFE sheet.

The microporous expanded PTFE layers were then secured at the ends of the mandrel to resist the tendency of this material to shrink back on itself at elevated temperatures. All the layers were then sintered while secured to the mandrel in an oven at 370°C for approximately 45 minutes to bond the layers together. After cooling, the material was spirally cut from the mandrel in the form of a continuous tape having a width of approximately 125 mm. The final thickness of the continuous tape was 4.8 mm. The final thickness of the full density expanded PTFE serving as the substantially air impermeable layer was 0.025 mm.

Example 5A

A 762 mm length of the tape from Example 5 was cut into sections of width of 29 mm. Layers of the microporous expanded PTFE sheet were removed from the tape to yield a tape having a thickness of 1.6 mm. A channel 44 as shown in Figure 1 was formed in the tape by making parallel cuts to the centerline of the length of the tape at a distance of 1.5 mm from the center. The depth of the cut was approximately 1.1 mm from the top of the tape where the full density PTFE film comprises the bottom of the tape. The microporous expanded PTFE material between the parallel cuts was peeled away and removed.

The tape was then folded in half as shown in Figure 2 resulting in a form-in-place gasket with a width of approximately 13 mm and a thickness of approximately 3.2 mm.

Example 5B

A section of length was cut from the continuous tape of Example 5. Layers of the microporous expanded PTFE sheet were peeled from the tape to yield a tape having a total thickness of 3.1 mm.

Comparative Example 6A

A continuous roll of 0.125 inch (3.2 mm) thick and 15 mm wide GORE-TEX ® BG Gasket Tape commercially available from W.L. Gore & Associates, Inc. of Newark, Delaware, was obtained. A length of 762 mm was cut from the roll.

Comparative Example 6B

A continuous roll of 0.125 inch (3.2 mm) thick and 75 mm wide GORE-TEX ® BG Gasket Tape commercially available from W.L. Gore & Associates, Inc. of Newark, Delaware, was obtained. A length of 2134 mm was cut from the roll.

Example 7

A form-in-place gasket of the present invention was produced in the following manner. A continuous expanded PTFE sheet produced from fine powder PTFE resin through paste-forming techniques was obtained and expanded in directions 90 degrees opposed to each other (longitudinally and transversely) to form a microporous expanded PTFE sheet as taught in US Patent Nos. 3,953,566 and 4,187,390 to Gore. This sheet, having a thickness of about 0.10 mm was then rolled between two rollers at a fixed gap to compress the microporous expanded PTFE sheet into a full density non-porous expanded PTFE sheet. This non-porous sheet had a final thickness of about 0.03 mm and a final width of about 1372 mm. This 1372 mm wide sheet was then slit into separate 150 mm wide sections that were taken up into separate rolls. One roll of this 150 mm wide sheet was used to spirally wrap around a 584 mm diameter mandrel so as to form a single layer thickness of the 0.03 mm film around the mandrel.

One hundred forty layers of the second microporous expanded PTFE sheet from Example 1, measuring approximately 1600 mm wide and 0.038 mm thick, was then wrapped on the mandrel covering the previously wrapped full density expanded PTFE sheet.

The microporous expanded PTFE layers were then secured at the ends of the mandrel to resist the tendency of this material to shrink back on itself at elevated temperatures. All the layers were then sintered while secured to the mandrel in an oven at 370°C for approximately 45 minutes to bond the layers together. After cooling, the material was spirally cut from the mandrel in the form of a continuous tape having a width of approximately 125 mm. The final thickness of the continuous tape was 4.8 mm. The final thickness of the full density expanded PTFE serving as the substantially air impermeable layer was 0.030 mm.

Example 7A

A 762 mm length of the tape from Example 7 was cut into sections of width of 29 mm. Layers of the microporous expanded PTFE sheet were removed from the tape to yield a tape having a total thickness of 1.6 mm. A channel 44 such as shown in Figure 1 was formed in the tape by making parallel cuts to the centerline of the length of the tape at a distance of 1.5 mm from the center. The depth of the cut was approximately 1.1 mm from the top of the tape where the full density PTFE film comprises the bottom of the tape. The microporous expanded PTFE material between the parallel cuts was peeled away and removed.

The tape was then folded in half as shown in Figure 2 resulting in a form-in-place gasket with a width of approximately 13 mm and a thickness of approximately 3.2 mm.

Example 7B

A section of length was cut from the continuous tape from Example 7. Layers of the microporous expanded PTFE sheet were peeled from the tape to yield a tape having a total thickness of 3.1 mm.

Example 7C

A 2134 mm length was cut from the continuous tape produced in Example 7. Layers of the microporous expanded PTFE sheet were peeled from the cut length of tape to yield a tape having a total thickness of 3.2 mm. This peeled tape was then slit to produce a tape having a width of 86 mm. A channel 44 such as shown in Figure 1 was formed in the 86 mm wide tape by making parallel cuts to the centerline of the length of the tape at a distance of 3.2 mm from the center. The depth of the cut was approximately 2.2 mm from the top of the tape where the full density PTFE film is at the bottom of the tape. The microporous expanded PTFE material between the parallel cuts was peeled away and removed.

The tape was then folded in half as shown in Figure 2 resulting in a form-in-place gasket with a width of approximately 40 mm and a thickness of approximately 6.4 mm.

Example 8

This example demonstrates a further embodiment of the present invention as shown in Figure 13 where a microporous expanded PTFE layer

161 is bounded on all four sides by an elastomer / expanded PTFE composite layer 163 serving as both the top and bottom substantially air impermeable layers 166a and 166b as well as forming the substantially air impermeable regions 165a and 165b on both the inside edge and outside edge.

First, a continuous roll of 0.125 inch (3.2 mm) thick and 20 mm wide GORE-TEX ® BG Gasket Tape commercially available from W.L. Gore & Associates, Inc. of Newark, Delaware, was obtained. A length of 762 mm was cut from this microporous expanded PTFE roll. The length of microporous expanded PTFE was then coated with a perfluoropolyether silicone elastomer, SIFEL™ 610, commercially available from Shin-Etsu Chemical Co., Ltd., in the following way. The length of material was dipped into a bath of the elastomer for a period of five minutes, allowing the elastomer to soak into the surface porosity of the GORE-TEX GR® BG Gasket Tape. Immediately after the five minutes of dipping, the excess elastomer was scraped from the surfaces of the tape. The coated tape was then cured in an oven at 175 °C for two hours producing the final form-in-place gasket 160. In this example, the elastomer / expanded PTFE composite layer 163 was approximately 0.13 mm thick.

Example 9

This example demonstrates a further embodiment of the present invention as shown in Figure 14 where a microporous expanded PTFE layer 151 is bounded only on one side edge and extending only partially across the top and bottom surfaces. Thus, the top and bottom air impermeable layers 156a and 156b and the air impermeable region 155 are formed from the elastomer / expanded PTFE composite.

First, a continuous roll of 0.125 inch (3.2 mm) thick and 20 mm wide GORE-TEX ® BG Gasket Tape commercially available from W.L. Gore & Associates, Inc. of Newark, Delaware, was obtained. A length of 762 mm was cut from this microporous expanded PTFE roll. The length of microporous expanded PTFE was then partially coated with a perfluoropolyether silicone elastomer, SIFEL™ 610, commercially available from Shin-Etsu Chemical Co., Ltd., in the following way. The length of material was partially dipped into a bath of the elastomer covering one side edge and partially covering the top and bottom surfaces for a period of five minutes, allowing the elastomer to soak into the surface porosity of the GORE-TEX GR® BG Gasket Tape. Immediately after the five minutes of dipping, the excess elastomer was scraped from the dipped surfaces of the tape. The coated tape was then cured in an oven at

175 °C for two hours producing the final form-in-place gasket 150. In this example, the elastomer / expanded PTFE composite layer 153 was approximately 0.13 mm thick.

Example 10

This example demonstrates a further embodiment of the present invention as shown in Figure 4 where a microporous expanded PTFE layer 61 is bounded on all four sides by a full density expanded PTFE layer 62 serving as both the top and bottom substantially air impermeable layers 66a and 66b as well as forming the substantially air impermeable regions 65a and 65b on both the inside edge and outside edge.

First, a length of the 0.03 mm thick unsintered non-porous full density expanded PTFE film from Example 7 was cut to a width of 50 mm. A 500 mm length of GORE-TEX ® BG Gasket Tape with a width of 40 mm and a thickness of 6.4 mm commercially available from W.L. Gore & Associates, Inc. of Newark, Delaware, was obtained. This tape is a microporous expanded PTFE tape. The 50 mm wide full density expanded PTFE film was helically wrapped around the GORE-TEX ® BG Gasket Tape at an angle to create a 6 mm wide overlap of the film. The wrapped tape was then pressed under light pressure in a laboratory press for a few seconds with platens heated to 365°C to bond the unsinterred full density expanded PTFE film to the microporous expanded PTFE tape. The tape was rotated 90 degrees and pressed again.

Example 11

This example demonstrates a further embodiment of the present invention as shown in Figure 4 where a microporous expanded PTFE layer 61 is bounded on all four sides by a composite PFA / full density expanded PTFE layer 62 serving as both the top and bottom substantially air impermeable layers 66a and 66b as well as forming the substantially air impermeable regions 65a and 65b on both the inside edge and outside edge.

First, a length of the 0.005 mm thick unsintered non-porous full density expanded PTFE film from Example 1 was cut to a width of 50 mm. A length of the 0.013 mm thick PFA film from Example 1 was also trimmed to a 50 mm width. A 500 mm length of GORE-TEX ® BG Gasket Tape from Example 10 with a width of 40 mm and a thickness of 6.4 mm was obtained. This tape is a microporous expanded PTFE tape. The PFA film was helically wrapped around the GORE-TEX ® BG Gasket Tape in the same type of wrapping

manner as was used in Example 10. The 0.005 mm thick full density expanded PTFE film was then helically wrapped around the PFA wrapped GORE-TEX ® BG Gasket Tape again using the same wrapping technique as was used in Example 10. The wrapped tape was then pressed under light pressure in the Carver laboratory press which was used in Example 10 for a few seconds with platens heated to 365°C to bond the unsinterred full density expanded PTFE film and the PFA film to the microporous expanded PTFE tape and to each other. The tape was rotated 90 degrees and pressed again.

Example 12

This example demonstrates a further embodiment of the present invention as shown in Figure 5 where gasket 70 is formed of a microporous expanded PTFE layer 71 bounded on three sides by a substantially air impermeable layer 72. Substantially air impermeable layer 72 can be alternatively described as comprising top and bottom substantially impermeable layers 76a and 76b bridged by substantially impermeable region 75. In this example, the substantially air impermeable layer 72 is a full density expanded PTFE layer.

First, a 500 mm length of the 0.03 mm thick unsintered non-porous full density expanded PTFE film from Example 7 was obtained. A 500 mm length of GORE-TEX ® BG Gasket Tape from Example 10 with a width of 40 mm and a thickness of 6.4 mm was obtained. This tape is a microporous expanded PTFE tape. The 0.03 mm thick full density expanded PTFE film was wrapped around three sides of the GORE-TEX ® BG Gasket Tape covering both top and bottom surfaces as well as one side edge. The wrapped tape was then pressed under light pressure in the Carver laboratory press which was used in Example 10 for a few seconds with platens heated to 365°C to bond the unsinterred full density expanded PTFE film to the microporous expanded PTFE tape. The tape was rotated 90 degrees and pressed again. The excess full density expanded PTFE film was trimmed and removed from the sample.

Example 13

This example demonstrates a further embodiment of the present invention as shown in Figure 5 where gasket 70 is formed of a microporous expanded PTFE layer 71 bounded on three sides by a substantially air impermeable layer 72. Substantially air impermeable layer 72 can be alternatively described as comprising top and bottom substantially

impermeable layers 76a and 76b bridged by substantially impermeable region 75. In this example, the substantially air impermeable layer 72 is a PFA / full density expanded PTFE composite layer.

First, a 500 mm length of the 0.005 mm thick unsintered non-porous full density expanded PTFE film from Example 1 was obtained. A 500 mm length of the 0.013 mm thick PFA film from Example 1 was obtained. A 500 mm length of GORE-TEX ® BG Gasket Tape from Example 10 with a width of 40 mm and a thickness of 6.4 mm was obtained. This tape is a microporous expanded PTFE tape. The PFA film was wrapped around three sides of the GORE-TEX ® BG Gasket Tape covering both top and bottom surfaces as well as one side edge. The 0.005 mm thick full density expanded PTFE film was then wrapped over the PFA film. The wrapped tape was then pressed under light pressure in the Carver laboratory press which was used in Example 10 for a few seconds with platens heated to 365°C to bond the unsinterred full density expanded PTFE film and the PFA film to the microporous expanded PTFE tape and to each other. The tape was rotated 90 degrees and pressed again. The excess full density expanded PTFE film and PFA film was trimmed and removed from the sample.

Sealability Test 1

Sealability was determined by leak rate tests performed in accordance with procedures and equipment outlined in ASTM F37-95 Test Method B, which is suitable for measuring precise leakage rates as high as 6 L/hr and as low as 0.3 ml/hr. The gasket stress was selected to be 10.3 MPa (1500 psi). The test fluid was air at 0.62 MPa (90 psi). The gaskets were formed into an annular shape using a skived cut overlap joint such as that shown in Figures 25-27 to join the ends. The excess length of the gaskets beyond what was needed to make the correct size annular shape with the skived cut overlap joint was discarded. The formed gaskets were then loaded to the selected compressive stress between two smooth steel press platens with a surface finish of RMS 32 held at room temperature. The gaskets were then subjected to the 0.62 MPa internal air pressure introduced into the center of the annular shaped formed gasket that was compressed between the press platens. The air pressure within the test assembly was then isolated from the environment by closing a valve. The leakage rate was determined by a change in the level of manometer fluid located in the line upstream from the gasket test fixture over a period of time. The change in the manometer was due to air leakage past

the gasket to the environment resulting in loss of internal air pressure. The manometer readings were converted to leakage rates using the equation below:

$$LR = \frac{MR * 2.54 * A * 60}{T * SG}$$

where: LR is Leakage Rate (ml/hr)
MR is manometer reading (inches)
2.54 constant is to convert manometer reading from (in) to (cm)
A is the cross sectional area of inside the manometer tube (cm^2)
T is time (min)
60 constant is to convert time from (min) to (hr)
SG is specific gravity of manometer fluid

The manometer linear scale must match the specific gravity of the fluid used. In this test, the manometer scale was calibrated for 0.827 specific gravity fluid. The fluid used was R 827 oil (specific gravity 0.827) commercially available from Dynatech Frontier Corporation of Albuquerque, New Mexico. The manometer used had an internal tube diameter of 0.25 inches (0.635 cm). Manometer readings were taken at five, ten and fifteen minutes.

The above volumetric leakage rate of air expressed in (ml/hr) can be further converted into a mass leakage rate of air in (mg/m*s), which is commonly used in the industry and which also takes into account the length around the perimeter of the gasket in the test. The leakage rate expressed in these terms is especially useful when for comparing leakage rates of gaskets having different perimeters. The following formula was used to calculate this mass leakage rate of air:

$$MLR = \frac{LR * D}{L * 3600}$$

where: MLR is mass leakage rate of air in (mg/m*s)
LR is volumetric leakage rate of air in (ml/hr)
D is density of air at ambient conditions in (mg/ml)
L is length around the perimeter of gasket in (m)
3600 constant is to convert time from (hr) to (s)

The density of air at ambient conditions in this test was assumed to be 1.2 mg/ml. The average of the inner and outer diameter of the formed gasket was used to compute the length of the perimeter around the formed gasket using the following equation:

$$L = \frac{\pi * (OD + ID)}{2}$$

where: L is length around the perimeter of the gasket in (m)
OD is the outside diameter of the formed gasket in (m)
ID is the inside diameter of the formed gasket in (m)

The sealability test above was conducted on the inventive embodiments of Examples 1A and 3A with the results shown in Table I below. The inner diameter of the formed gaskets in this test was 0.1524 m. The outer diameter of the formed gaskets in this test was 0.1778 m. The average diameter of the formed gaskets was therefore 0.1651 m, giving a computed length around the perimeter of the formed gasket of 0.5187 m. The results show that both of these inventive examples had a very low leak rate, in all cases measuring below 0.001 mg/(m*s). In the case of Example 1A, the leak rate was extremely low in that the leak rate was less than 0.00001 mg/m*s.

Table I

Example 1A

Run #1

S.G. of Manometer fluid = 0.827

Reading	Time (min)	Manometer Reading (in H2O)	LeakRate (mL/hr)	LeakRate Mg/(m*s)
1	5	0.0	0.00	0.00000
2	10	0.0	0.00	0.00000
3	15	0.0	0.00	0.00000
		Average Leak Rate:	0.00	0.00000

Example 3A

Run#1

S.G. of Manometer fluid = 0.827

Reading	Time (min)	Manometer Reading (in H2O)	LeakRate (mL/hr)	LeakRate mg/(m*s)
1	5	0.00	0.00	0.00000
2	10	0.03	0.18	0.00011
3	15	0.03	0.12	0.00008
		Average Leak Rate:	0.10	0.00006

Example 1A
Run #2
S.G. of Manometer fluid = 0.827

Reading	Time (min)	Manometer Reading (in H2O)	LeakRate (mL/hr)	LeakRate Mg/(m*s)
1	5	0.0	0.00	0.00000
2	10	0.0	0.00	0.00000
3	15	0.0	0.00	0.00000
		Average Leak Rate:	0.00	0.00000

Example 3A
Run #2
S.G. of Manometer fluid = 0.827

Reading	Time (min)	Manometer Reading (in H2O)	LeakRate (mL/hr)	LeakRate mg/(m*s)
1	5	0.10	1.17	0.00075
2	10	0.20	1.17	0.00075
3	15	0.30	1.17	0.00075
		Average Leak Rate:	1.17	0.00075

Example 1A
Run #3
S.G. of Manometer fluid = 0.827

Reading	Time (min)	Manometer Reading (in H2O)	LeakRate (mL/hr)	LeakRate Mg/(m*s)
1	5	0.0	0.00	0.00
2	10	0.0	0.00	0.00
3	15	0.0	0.00	0.00
		Average Leak Rate:	0.00	0.00000

Example 3A
Run #3
S.G. of Manometer fluid = 0.827

Reading	Time (min)	Manometer Reading (in H2O)	LeakRate (mL/hr)	LeakRate mg/(m*s)
1	5	0.05	0.58	0.00038
2	10	0.10	0.58	0.00038
3	15	0.20	0.78	0.00050
		Average Leak Rate:	0.65	0.00042

Sealability Test 2

This sealability test was conducted exactly like Sealability Test 1 above except that the manometer fluid used was Meriam 100 Unity Oil (specific gravity 1.00) commercially available from Meriam Instrument, a division of the Scott & Fetzer Co., of Cleveland, Ohio. The manometer scale used was calibrated for 1.00 specific gravity fluid. The test fluid, again, was air at 0.62 MPa (90 psi). In this test, the manometer readings were taken at various time intervals for the different gaskets up to 60 minutes, and a leak rate was computed for each time interval measured.

This sealability test was conducted on the inventive embodiments of Examples 3A and 5A, and on Comparative Examples 2A and 6A with the results shown in Table II below. The average leak rate for each sample type in this test as well as the average leakage rate for each sample type of Sealability Test I is graphed in Figure 24. For the case of Example 3A, which had samples run in both tests, the leakage rate was averaged between the two tests.

The same size gaskets were formed as in Sealability Test 1. The same skived cut overlap joint technique was used for Examples 3A and 5A as was used in Sealability Test 1 and illustrated in Figures 25-27. A skived cut overlap joint such as illustrated in Figures 22 and 22A was used for Comparative Example 6A. Comparative Example 2A was cut from a sheet to the specified size and thus, did not have to be formed and did not have a joint.

It can be seen from the results in Table I, Table II and the graph of Figure 24 that the lowest leak rate (best performance) was achieved by the inventive embodiment of Example 1A, followed by the inventive embodiment of Example 3A. Both of these inventive examples had a much lower leakage rate than both of the comparative examples. Example 1A had a leak rate improvement of more than three orders of magnitude over the comparative examples. Example 3A had a leak rate improvement of more than one order of magnitude over the comparative examples. The inventive embodiment of Example 5A represented a much smaller improvement in leak rate over the comparative examples.

Table II

Example 3A
Run #1
S.G. of Manometer fluid = 1.00

Reading	Time (min)	Manometer Reading (in H2O)	LeakRate (mL/hr)	LeakRate mg/(m*s)
1	2.5	0	0.00	0.00000
2	5	0	0.00	0.00000
3	10	0	0.00	0.00000
4	15	0.05	0.16	0.00010
Average Leak Rate:		0.04	0.00003	

Example 3A
Run #2
S.G. of Manometer fluid = 1.00

Reading	Time (min)	Manometer Reading (in H2O)	LeakRate (mL/hr)	LeakRate mg/(m*s)
1	5	0	0.00	0.00000
2	10	0.05	0.24	0.00016
3	15	0.08	0.26	0.00017
		Average Leak Rate:	0.17	0.00011

Example 3A

Run #3

S.G. of Manometer fluid = 1.00

Reading	Time (min)	Manometer Reading (in H2O)	LeakRate (mL/hr)	LeakRate mg/(m*s)
1	2.5	0	0.00	0.00000
2	5	0	0.00	0.00000
3	10	0	0.00	0.00000
4	15	0	0.00	0.00000
5	20	0	0.00	0.00000
6	30	0	0.00	0.00000
7	60	0.2	0.16	0.00010
		Average Leak Rate:	0.02	0.000015

Example 5A

Run #1

S.G. of Manometer fluid = 1.00

Reading	Time (min)	Manometer Reading (in H2O)	LeakRate (mL/hr)	LeakRate mg/(m*s)
1	2.5	0.8	15.44	0.00993
2	5	1.4	13.51	0.00868
3	7.5	2.1	13.51	0.00868
		Average Leak Rate:	14.16	0.00910

Example 5A

Run #2

S.G. of Manometer fluid = 1.00

Reading	Time (min)	Manometer Reading (in H2O)	LeakRate (mL/hr)	LeakRate mg/(m*s)
1	2.5	1.0	19.31	0.01241
2	5.0	2.0	19.31	0.01241
3	7.5	3.0	19.31	0.01241
		Average Leak Rate:	19.31	0.012406

Example 5A

Run #3

S.G. of Manometer fluid = 1.00

Reading	Time (min)	Manometer Reading (in H2O)	LeakRate (mL/hr)	LeakRate mg/(m*s)
1	2.5	1.5	28.96	0.01861
2	5.0	2.7	26.06	0.01675
3	7.5	4.1	26.38	0.01696
		Average Leak Rate:	27.14	0.01744

Comparative Example 2A
Run #1
S.G. of Manometer fluid = 1.00

Reading	Time (min)	Manometer Reading (in H2O)	LeakRate (mL/hr)	LeakRate mg/(m*s)
1	5	2.4	23.17	0.01489
2	7	3.4	23.44	0.01506
		Average Leak Rate:	23.30	0.01498

Comparative Example 2A
Run #2
S.G. of Manometer fluid = 1.00

Reading	Time (min)	Manometer Reading (in H2O)	LeakRate (mL/hr)	LeakRate mg/(m*s)
1	2.5	1.5	28.96	0.01861
2	5.0	2.9	27.99	0.01799
3	7.5	4.3	27.67	0.01778
		Average Leak Rate:	28.21	0.01813

Comparative Example 6A
Run #1
S.G. of Manometer fluid = 1.00

Reading	Time (min)	Manometer Reading (in H2O)	LeakRate (mL/hr)	LeakRate mg/(m*s)
1	2.5	1.4	27.03	0.01786
2	5	2.2	21.24	0.01403
3	7.5	4.1	26.38	0.01743
		Average Leak Rate:	24.88	0.01644

Comparative Example 6A
Run #2
S.G. of Manometer fluid = 1.00

Reading	Time (min)	Manometer Reading (in H2O)	LeakRate (mL/hr)	LeakRate mg/(m*s)
1	2.5	0.8	15.44	0.01020
2	5.0	1.6	15.44	0.01020
3	7.5	2.3	14.80	0.00978
		Average Leak Rate:	15.23	0.01006

Sealability Test 3 (Bubble test)

Another type of sealability test, representing what is known in the industry as a "bubble test", was performed which involved checking for air leakage from a gasketed glass-lined steel flanged pipe section using soapy water. A cross sectional view of the bubble test fixture used to perform this test is shown in Figure 16. The bubble test results are shown in Table III below. The test fixture 100 was constructed from two 610 mm diameter glass lined steel flanged pipe sections 101 and 102. Steel plates 103 and 104 were welded to the non-flanged ends of the pipe sections 101 and 102 to create a closed vessel when the flanged ends are clamped together. The two pipe sections 101 and 102 are clamped together using 7/8" J-Type clamps 105. The gasket 106 to be tested was installed on the flange of the lower pipe section 101. The upper pipe section 102 was positioned above the lower pipe section 101 and the clamps 105 were installed.

Twenty clamps 105 were evenly spaced around the perimeter of the test fixture 100. The clamps 105 were tightened in a crossing-type pattern in three passes using a torque wrench at torque levels of 40.7 N-m (30 ft-lbs), 81.3 N-m (60 ft-lbs) and finally 122.0 N-m (90 ft-lbs). Two circular torque patterns were done at 122.0 N-m after the cross pattern at 122.0 N-m. The clamps were re-tightened to 122.0 N-m in a circular pattern after waiting 30 minutes after the second circular pattern.

The vessel was then pressurized with air through an air regulator 107 to the first pressure level of 0.21 MPa (30 psi) using a pressure gauge 108 to measure the pressure level. The gasket 106 was checked for leaks by first, spraying the outside perimeter of the gasket with the soapy water solution. The gasket 106 was then visually checked for bubbles appearing in the soapy water along the outer diameter of the gasket 106 indicating air leakage. After determining whether or not there were air bubbles present, the internal air pressure was increased to the next level of 0.41 MPa (60 psi). Again, after determining whether or not there were air bubbles present at this pressure level, the internal air pressure was increased to the final level of 0.62 MPa (90 psi), where once again it was determined whether or not air bubbles were present. The internal air pressure was then released and the gasket was removed.

This bubble test was conducted on the inventive embodiments of gaskets produced in accordance with Examples 1C and 7C as well as on gaskets from Comparative Examples 2B and 6B. Examples 1C and 7C and Comparative

Example 6B were formed into an annular shape fitting the flange using a skived cut overlap joint to join the ends. Examples 1C and 7C used a skived cut overlap joint such as that shown in Figures 25-27. Comparative Example 6B used a skived cut overlap joint such as that shown in Figures 22 and 22A. The excess length of the gaskets beyond what was needed to make the correct size annular shape with the skived cut overlap joint was discarded. Comparative Example 2B was cut from a sheet to the specified size and thus, did not have to be formed and did not have a joint.

The results are shown in Table III below. The test results demonstrate the improved sealability of the inventive gaskets from Examples 1C and 7C over that of the conventional microporous expanded PTFE gaskets represented by Comparative Examples 2B and 6B, as evidenced by the absence of any air bubbles in any of the test conditions for the inventive gaskets. The conventional microporous expanded PTFE gaskets showed bubbles, indicating leakage in all of the tested conditions. In looking at the boundary conditions of the test, while the conventional gaskets showed leakage at the least demanding test condition (0.21 MPa internal pressure), the inventive gaskets showed no leakage at even the most demanding test condition (0.62 MPa internal pressure). This demonstrates a vast improvement of sealability of the inventive gaskets over the conventional microporous expanded PTFE gaskets.

Table III

	Air Pressure (MPa)	Bubbles Detected
Example 1C	0.21	No
	0.41	No
	0.62	No
Example 7C	0.21	No
	0.41	No
	0.62	No
Comparative	0.21	Yes
Example 2B	0.41	Yes
	0.62	Yes
Comparative	0.21	Yes
Example 6B	0.41	Yes
	0.62	Yes

Air Permeability Test 4

As a means of measuring the air permeability level, and consequently air impermeability level, of various film or sheet materials, a test fixture having an overall internal air volume of 50 cc was constructed. This air impermeable test fixture is shown in Figure 17. The air permeability test fixture 120 was created using a 1.5 inch (3.81 cm) diameter sanitary flange ferrule 121. The ferrule 121 was cut to a length of 5.2 cm and welded to a stainless steel base 122. A hole 123 was drilled through the base for connection to a pressurized air source and pressure measurement instrumentation. All components of the test fixture 120 were connected using 1/8 inch tubing and compression fittings. A digital manometer 124 (350 Smart Manometer commercially available from Meriam Instrument of Cleveland, Ohio) was used to accurately measure pressure. A regulated air supply was used to pressurize the test fixture to the proper starting pressure. A shut off valve 126, connected with compression fittings, was used to block airflow to or from the test fixture once the desired internal pressure was achieved. The overall internal air volume of the test fixture 120 was based on the internal air volume of the fixture 120 including the volume associated with fittings and tubing sections between the shut-off valve 126 and the interior of the flange ferrule 121. The total fixture volume (chamber + volume in tubing and fittings) was calculated to be 50 cubic centimeters (± 0.5 cc).

To test a film or sheet sample 127, the sample 127 was cut into a circle having a diameter of 5.1 mm (2.0 inches). The film 127 was placed over the opening of the sanitary flange ferrule 121. A 1.5 inch (3.81 cm) diameter screened EPDM gasket 128, having a stainless steel screen with a mesh size of 40 bounded around the perimeter by EPDM rubber commercially available from Rubberfab Mold and Gasket Co. of Andover, New Jersey, under part number 40MP-ES150, was placed on top of the test sample 127 to serve as a backing to keep the test film 127 from distending and/or bursting during the test. A 1.5 inch (3.81 cm) short weld sanitary flange ferrule 129 was placed on top of the screened EPDM gasket 128 and the sanitary flange clamp 125 was tightened into place, creating a seal between the flange ferrule 121, the film sample 127, the screened EPDM gasket 128, and the short weld sanitary flange ferrule 129. The regulated air supply connected to the valve 126 was used to create the initial internal pressure of the test fixture 120. The fixture 120 was pressurized to a pressure of 50.0 kPa and the valve 126 was closed. A stopwatch was used to measure the time required for the pressure within the

test fixture 120 to drop from 50.0 kPa to10.0 kPa as a result of air permeation through the film test sample 127. For highly impermeable film samples (where the internal fixture pressure requires greater than ten minutes to fall from 50.0 kPa to 10.0 kPa) the pressure was recorded after 10 minutes. Table IV below shows the air impermeability results using the test procedures described above for various film type samples. Three test samples were made and tested for each film type sample. The following film type samples were tested.

Film Type Sample A - This 0.03 mm thick nonporous (full density) expanded PTFE film was produced by peeling the nonporous full density expanded PTFE layer from a section of the continuous tape that was spirally cut from the mandrel in Example 7. Three circles having a diameter of 5.1 mm were cut from this film to produce the test samples.

Film Type Sample B - This 0.03 mm thick nonporous (full density) expanded PTFE film was produced by peeling the nonporous full density expanded PTFE layer from a section of the continuous tape that was spirally cut from the mandrel in Example 3. Three circles having a diameter of 5.1 mm were cut from this film to produce the test samples.

Film Type Sample C - This 0.015 mm thick nonporous (full density) expanded PTFE film was produced by peeling the nonporous expanded PTFE outer layers from the sheet that was cut from the mandrel in Example 4. Three circles having a diameter of 5.1 mm were cut from this film to produce the test samples.

Film Type Sample D - This 0.025 mm thick nonporous (full density) expanded PTFE film was produced by peeling the nonporous expanded PTFE outer layers from the sheet that was cut from the mandrel in Example 5. Three circles having a diameter of 5.1 mm were cut from this film to produce the test samples.

Film Type Sample E – The 0.051 mm thick skived PTFE was from a roll of full density skived PTFE (0.051 mm thick, 610 mm wide) commercially available from Fluoroplastics, Inc., of Philadelphia, Pennsylvania. Three circles having a diameter of 5.1 mm were cut from this film to produce the test samples.

Film Type Sample F – The 0.051 mm thick PFA film was obtained and is commercially available from E.I. du Pont de Nemours, Inc., of Wilmington, Delaware, under part number 200LP high performance PFA film. Three circles having a diameter of 5.1 mm were cut from this film to produce the test samples.

Film Type Sample G – The 0.013 mm thick PFA film was from the commercially available PFA film from Example 1. Three circles having a diameter of 5.1 mm were cut from this film to produce the test samples.

Film Type Sample H - The 0.038 mm thick microporous expanded PTFE film was from the second continuous microporous expanded PTFE sheet produced in Example 1. Three circles having a diameter of 5.1 mm were cut from this film to produce the test samples.

Film Type Sample I – The 3.2 mm thick GORE-TEX® GR Sheet was from a sheet of commercially available GORE-TEX® GR Sheet gasketing such as that used in Comparative Example 2. This is a microporous expanded PTFE sheet gasketing material. Three circles having a diameter of 5.1 mm were cut from this film or sheet to produce the test samples.

Film Type Sample J – The 1.0 mm thick GORE-TEX® GR Sheet was obtained and is commercially available from W. L. Gore and Associates, Inc. This is a microporous expanded PTFE sheet gasketing material. Three circles having a diameter of 5.1 mm were cut from this film or sheet to produce the test samples.

Film Type Sample K - The 3.0 mm thick microporous expanded PTFE film was produced by peeling the outer full density PTFE layers from the sheet which was cut from the mandrel in Example 1, leaving the inner microporous expanded PTFE layer. Three circles having a diameter of 5.1 mm were cut from this microporous expanded PTFE film (layer) to produce the test samples.

Table IV

Film Type Sample	Film Sample Construction	Test Sample	Time for Test Completion (seconds)
A	Full density expanded PTFE 0.03 mm thickness	1	600* (49.6 kPa)
		2	600* (49.4 kPa)
		3	600* (48.8 kPa)
B	Full density expanded PTFE 0.03 mm thickness	1	600 * (32.8 kPa)
		2	600* (30.0 kPa)
		3	600 * (22.7 kPa)
C	Full density expanded PTFE 0.015 mm thickness	1	600* (13.8 kPa)
		2	600* (12.9 kPa)
		3	535.13
D	Full density expanded PTFE 0.025 mm thickness	1	20.8
		2	26.0
		3	18.9
E	Skived PTFE 0.051 mm thickness	1	600* (49.9 kPa)
		2	600* (49.8 kPa)
		3	600* (49.8 kPa)
F	PFA film 0.051 mm thickness	1	600* (49.9 kPa)
		2	600* (49.9 kPa)
		3	600* (49.8 kPa)
G	PFA film 0.013 mm thickness	1	600* (49.9 kPa)
		2	600* (49.9 kPa)
		3	600* (49.9 kPa)
H	Microporous expanded PTFE 0.038 mm thickness	1	0.5
		2	0.4
		3	0.5
I	Microporous expanded PTFE 3.2 mm thickness	1	5.5
		2	5.5
		3	5.5
J	Microporous expanded PTFE 1.0 mm thickness	1	1.9
		2	1.8
		3	1.8
K	Microporous expanded PTFE 3.0 mm thickness	1	5.6
		2	5.5
		3	5.3

From observing the test results it can be seen that all of the represented materials which were used in the inventive examples for substantially air impermeable layers and those materials which could be used as effective substantially air impermeable layers were more air impermeable than the materials used representing the microporous expanded PTFE inner layers. This is evident because of the longer amount of time it took the film type samples representing the substantially air impermeable layers to drop from 50.0 kPa to10.0 kPa (or in the cases where the 600 second time limit was reached, the less pressure was lost) as compared to those film type samples representing the microporous expanded PTFE inner layers. Film type samples A, B, C, D and G represented some of the different materials used in the inventive examples as substantially air impermeable layers. Film type samples

E and F represented other materials which could be used as effective substantially air impermeable layers. Film type sample K represented a material used in the inventive examples as the microporous expanded PTFE inner layer. Film type sample H represented a single layer of the microporous expanded PTFE film which was used to generate the microporous expanded PTFE inner layer of some of the inventive examples. Film type examples I and J represented commercially available microporous expanded PTFE sheet gasketing which could be used effectively as the microporous expanded PTFE inner layer.

It can also be seen from these results that within the groupings of similar materials that are differentiated by their thickness levels, generally, the thicker the material the more air impermeable it becomes, as evident by the longer time it takes for the pressure level to drop (or in the cases where the 600 second time limit was reached, the less pressure was lost) . In comparing the different levels of thickness of the similar densified expanded PTFE materials of Film Type Samples A, B, C and D, the increasing level of thickness of the material generally showed an increasing level of air impermeability. One exception to this trend was with Film Type Sample D which was not as air impermeable as Film Type Sample C. It is believed that the level of full density for Film Type Sample D was not as high as the level of full density achieved in Film Type Sample C, thus explaining the lower level of air impermeability exhibited by Film Type Sample D. In comparing the different levels of thickness of the similar microporous expanded PTFE materials of Film Type Samples H, I, J and K, once again, the thicker the material, the more air impermeable it was. In the case of the two PFA film samples of Film Type Sample F and G, both samples were extremely air impermeable in that after the 600 seconds (ten minutes) the air pressure had only dropped from 50 kPa to 48.8 - 49.9 kPa.

It can also be seen from these results that the film type samples representing the materials used in the inventive examples as the substantially air impermeable layers as well as the samples representing materials which could be effectively used as substantially air impermeable layers (Film Type Samples A , B, C, D, E, F and G) were all much thinner than the film type samples representing the materials used as the microporous expanded PTFE inner layer or which could be effectively used as the microporous expanded PTFE inner layer (Film Type Samples I, J and K). As previously mentioned, it can be advantageous to use materials which are highly air impermeable at

relatively low levels of thickness to enhance the conformability of the final gasket. Thus, it is demonstrated that full density expanded PTFE (or near full density expanded PTFE), PFA films, and skived PTFE are all materials that can be effectively used as the substantially air impermeable layer.

By combining the results from Sealability Test 1 and the results from this test, It has been further demonstrated that materials with results in this test of equal to or greater than about 20 seconds can be useful as a substantially air impermeable layer. It has also been shown that materials with results in this test greater than about 100 seconds can be even more effective as a substantially air impermeable layer. It has further been shown that materials with results in this test greater than 600 seconds can be most effective as a substantially air impermeable layer.

Air Permeability Test 5

The same equipment and procedure was used for this test as was done in the preceding Air Permeability Test 4 except that this time the screened EPDM gasket 128 which served as backing material was not used. All of the samples measured in this test were thick enough such that the extra backing that the screened EPDM gasket 128 supplied was not needed. As in the preceding test, three circles having a diameter of 5.1 mm were cut from the film or sheet to be tested to produce the test samples. For Comparative Example 2 and the inventive embodiment of Example 4 the time was measured and recorded for the pressure within the test fixture 120 to drop from 50.0 kPa to10.0 kPa as a result of air permeation through the test sample 127. For the much more highly air impermeable test samples 127 from the inventive embodiments of Examples 1B and 3B, the time was measured and recorded for the pressure to drop to 49.5 kPa and 48 kPa respectively. A comparative leak rate in terms of pressure drop per area over time was calculated for each test sample 127 using the following equation:

$$\text{Leak Rate} = \frac{(IP - FP)}{A * T}$$

where: Leak Rate is in (kPA / cm^2 / sec)
IP is initial pressure in (kPa)
FP is final pressure in (kPa)
A is area of test sample exposed to test pressure in (cm^2)
T is time in (sec)

For all of the test samples 127 measured, the area of the test sample exposed to test pressure was 9.58 cm^2, which was the calculated area inside the 1.5 inch (3.81 cm) diameter sanitary flange ferrule 121. The inside diameter of this sanitary flange ferrule 121 was 3.4925 cm.

All of the data recorded for Air Permeability Test 5 was recorded in Table V below. By observing both the time measurements for the various pressure drops and the calculated leak rate measurements, it can be seen that all of the samples of the inventive embodiments represented by Examples 1B, 3B and 4 were much for air impermeable than the conventional microporous expanded PTFE material of Comparative Example 2, as evidenced by the longer time periods it took to lose pressure and by the lower comparative leak rate calculations. Example 4 had a calculated leak rate improvement of more than one order of magnitude less than Comparative Example 2. Example 3B had a calculated leak rate improvement more than two orders of magnitude less than Comparative Example 2. Example 1B had a calculated leak rate improvement more than three orders of magnitude less than Comparative Example 2. The determining difference between the inventive embodiments of Examples 1B, 3B and 4 and Comparative Example 2 was the substantially air impermeable layer portion of the inventive examples. Both the inventive examples and the comparative example had a similar thickness of microporous expanded PTFE to permeate through, however the inventive examples had the additional substantially air impermeable layer which first had to be permeated before air could permeate through the microporous expanded PTFE.

Table V

Sample ID	Run #	Thickness (inches)	Thickness (mm)	Initial Pressure (kPa)	Final Pressure (kPa)	Time (seconds)	Leak Rate ($kPa/cm^2/sec$)
Comparative	1	0.119	3.02	50	10	5.06	0.8252
Example 2	2	0.119	3.02	50	10	5.82	0.7174
	3	0.121	3.07	50	10	5.74	0.7274
	Average	**0.120**	**3.04**	**50**	**10**	**5.54**	**0.7567**
Example 4	1	0.118	3.00	50	10	73.65	0.0567
	2	0.117	2.97	50	10	96.1	0.0434
	3	0.116	2.95	50	10	108.58	0.0385
	Average	**0.117**	**2.97**	**50**	**10**	**92.78**	**0.0462**
Example 1B	1	0.116	2.95	50	49.5	464.33	0.0001
	2	0.116	2.95	50	49.5	532.3	0.0001
	3	0.119	3.02	50	49.5	280.49	0.0002
	Average	**0.117**	**2.97**	**50**	**49.5**	**425.71**	**0.0001**
Example 3B	1	0.123	3.12	50	48	61.44	0.0034
	2	0.121	3.07	50	48	50.72	0.0041
	3	0.121	3.07	50	48	42.42	0.0049
	Average	**0.122**	**3.09**	**50**	**48**	**51.53**	**0.0041**

Solvent Permeation Test 6

To measure the permeation of a solvent into a gasket material, the test fixture 130 shown in Figure 21 was used. The fixture 130 was comprised of a top threaded glass jar 131, a metal washer 132, and a threaded, open center lid 133. The test sample 134 was prepared by cutting a disc having a diameter of 68.5 mm from a section of the gasket material to be tested. An ethyl alcohol based red ink, part number 5311 commercially available from Imaje Ink Jet Printing Corporation of Smyrna, Georgia was used as the solvent. First, the glass jar 131 was approximately half filled with the ink. The test sample 134 was then placed on top of the jar with the surface to be tested for permeability facing down. The metal washer 132 was placed inside the lid 133 and the lid 133 was secured to the jar 131 by screwing the lid 133 onto the jar 131. The lid 133 was firmly hand tightened to prevent the solvent from leaking around the

test sample 134 in the jar 131. The jar 131 was then inverted so that the solvent was in direct contact with the surface to be tested of the test sample 134. The jar 131 remained inverted for two hours. After the two hours of exposure, the test sample 134 was removed from the jar 131. The surface of the test sample 134 in contact with the ink was rinsed with a clean solvent to remove any residual ink. The test sample 134 was allowed to dry for approximately 30 minutes.

After drying, the test sample 134 was cut in half down the diameter of the sample such that the inner cross section of the test sample 134 could be easily viewed. This cross sectional view inside the test sample 134 is depicted in Figure 21A. Permeation of the ink into the test sample 134 is identifiable by the red coloration 136 in the thickness of the sample. The depth of this penetration into the thickness of the test sample 134 as well as the overall thickness of the test sample 134 was measured and recorded in Table VI below.

The inventive embodiments of Examples 1B, 3B, 4B, 5B and 7B were tested as well as Comparative Examples 2A and 6A. From observing the data in table VI, it can be seen that all of the inventive examples had no penetration of the solvent into the test sample. The comparative examples, on the other hand, showed a large amount of solvent penetration into the test samples. This demonstrates a vast improvement of the inventive gaskets over the conventional comparative gaskets to the resistance to liquid permeation through the gasket. Furthermore, this test demonstrates the effectiveness of the substantially air impermeable layers of these inventive examples to also be very effective against liquid permeation, even low surface tension liquids such as solvents.

Table VI

SAMPLE	SAMPLE THICKNESS	DEPTH OF INK PERMEATION
Example 1B	3 mm	0 mm
Example 3B	3 mm	0 mm
Example 4B	3 mm	0 mm
Example 5B	3 mm	0 mm
Example 7B	3 mm	0 mm
Comparative Example 2A	3 mm	1.45mm
Comparative Example 6A	3 mm	1.70 mm

While particular embodiments of the present invention have been illustrated and described herein, the present invention should not be limited to such illustrations and descriptions. It should be apparent that the changes and modifications may be incorporated and embodied as part of the present invention within the scope of the following claims.

The invention claimed is:

1. A multilayer, unitary gasket comprising:

at least one inner layer of expanded PTFE disposed between

a first substantially air impermeable outer layer and

a second substantially air impermeable outer layer, and

a substantially air impermeable region bridging said first and second substantially air impermeable layers.;

wherein said gasket is a form-in-place gasket.

2. A multilayer, unitary gasket as defined in claim 1 wherein said at least one inner layer of expanded PTFE has an inside edge and an outside edge.

3. A multilayer, unitary gasket as defined in claim 2 wherein said substantially air impermeable region is disposed on said inside edge.

4. A multilayer, unitary gasket as defined in claim 2 wherein said substantially air impermeable region is disposed on said outside edge.

5. A multilayer, unitary gasket as defined in claim 2 wherein said substantially air impermeable region is disposed between said inside edge and said outside edge.

6. A multilayer, unitary gasket as defined in claim 1 wherein said substantially air impermeable region comprises densified expanded PTFE.

7. A multilayer, unitary gasket as defined in claim 1 wherein said substantially air impermeable region comprises expanded PTFE having a structure of interconnected passages and pathways and a filler disposed in at least a portion of said passages and pathways.

8. A multilayer, unitary gasket as defined in claim 7 wherein said filler is an elastomer.

9. A multilayer, unitary gasket as defined in claim 7 wherein said filler is a fluoroelastomer.

10. A multilayer, unitary gasket as defined in claim 7 wherein said filler is a perfluoroelastomer.

11. A multilayer, unitary gasket as defined in claim 7 wherein said filler is a perfluoropolyether silicone elastomer.

12. A multilayer, unitary gasket as defined in claim 1 wherein said first and second substantially air impermeable layers comprise densified expanded PTFE.

13. A multilayer, unitary gasket as defined in claim 1 wherein said first and second substantially air impermeable layers comprise skived PTFE.

14. A multilayer, unitary gasket as defined in claim 1 wherein said first and second substantially air impermeable layers comprise PTFE.

15. A multilayer, unitary gasket as defined in claim 1 wherein said first and second substantially air impermeable layers are selected from the group consisting of PFA and FEP.

16. A multilayer, unitary gasket as defined in claim 1 wherein there are at least two of said inner layers of expanded PTFE and further comprising a substantially air impermeable layer disposed between said at least two inner layers.

17. A multilayer, unitary gasket as defined in claim 1 further comprising a plurality of said substantially air impermeable regions.

18. A method of making a form-in-place gasket comprising the steps of:

(a) providing at least one layer of expanded PTFE;

(b) bonding a substantially air impermeable layer to said layer of expanded PTFE to form a multilayer, unitary composite;

(c) removing a portion of said layer of expanded PTFE over said air impermeable layer to form a channel; and

(d) folding said multilayer unitary composite around said channel.

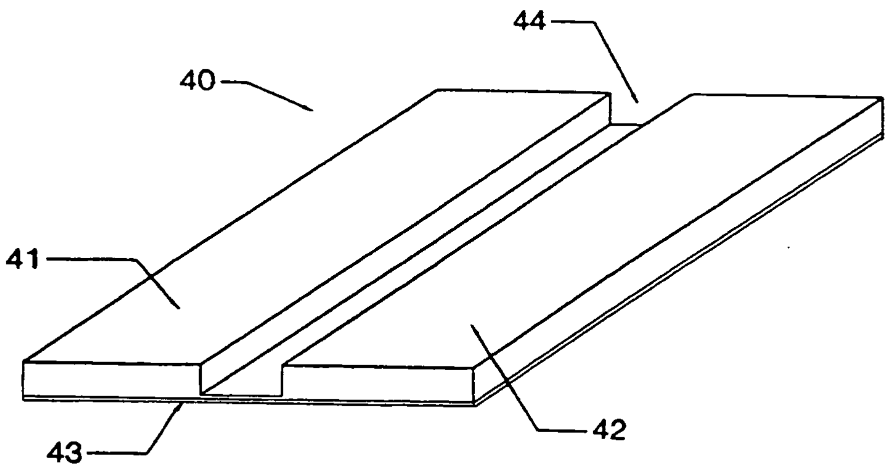


FIG. 1

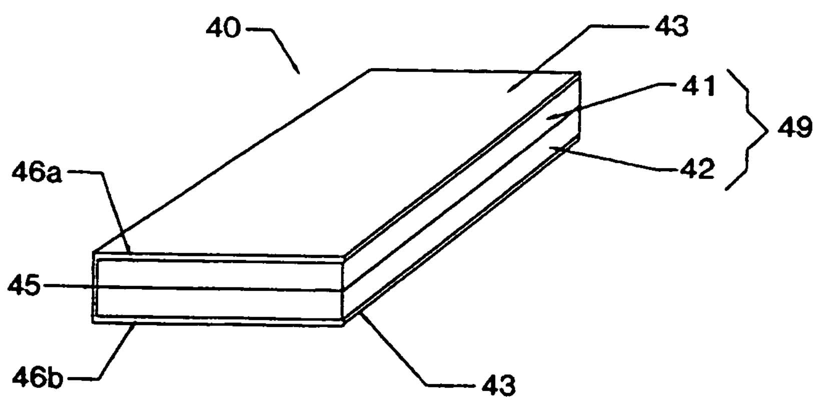


FIG. 2

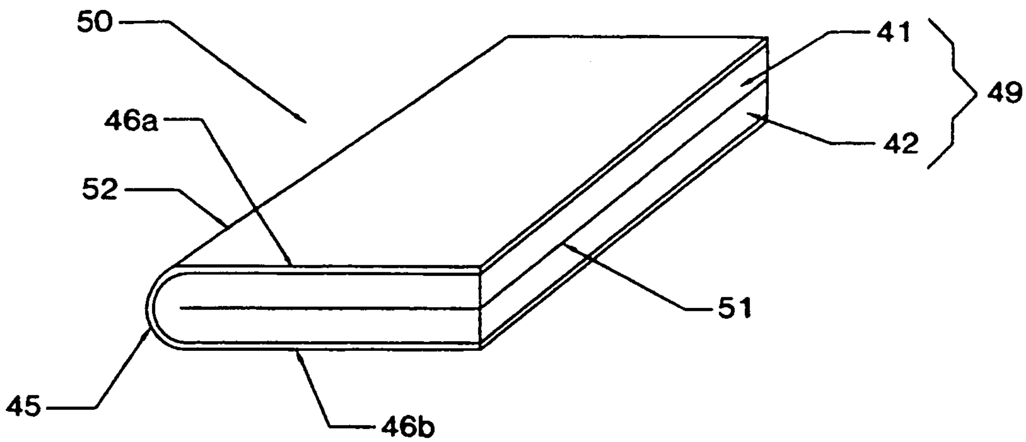


FIG. 3

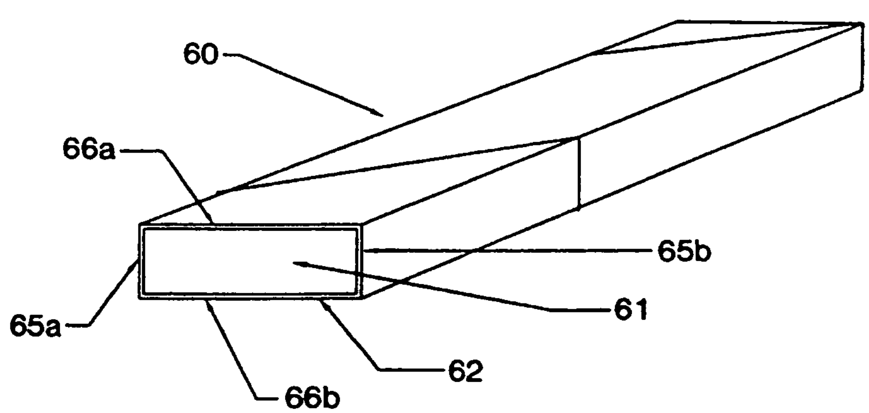


FIG. 4

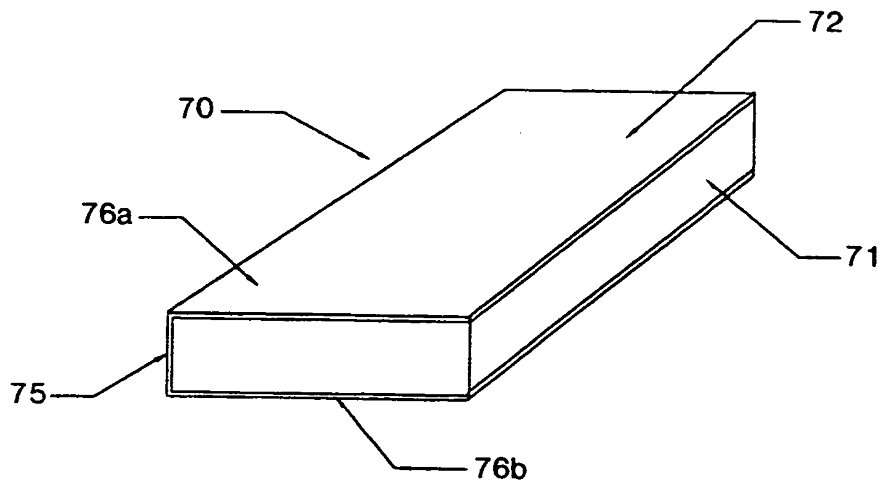


FIG. 5

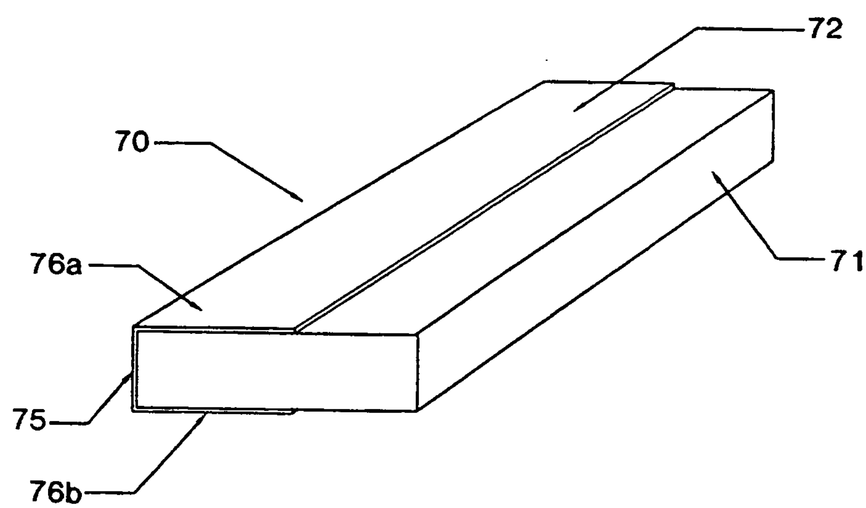


FIG. 6

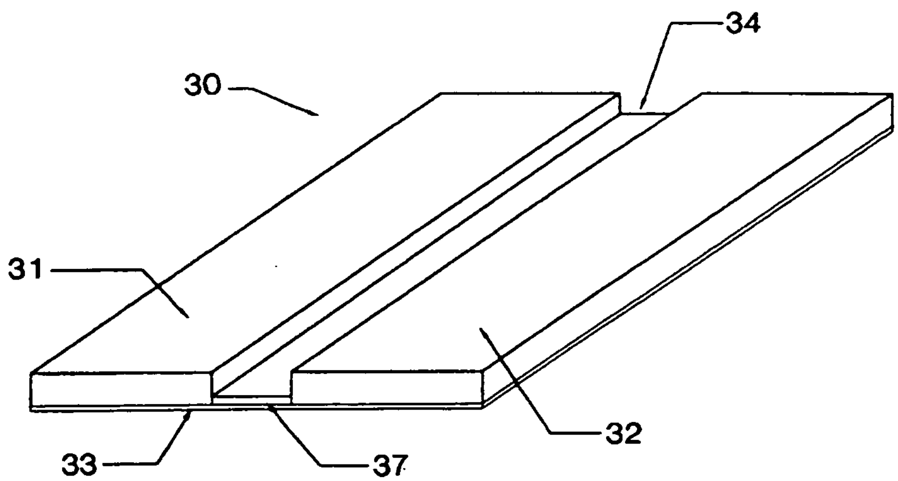


FIG. 7

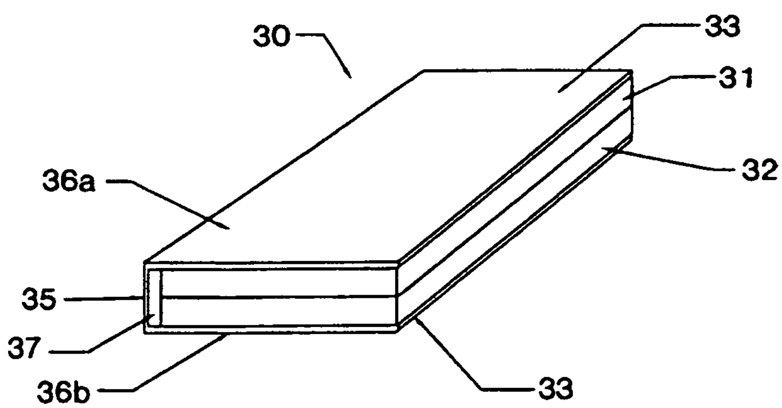


FIG. 8

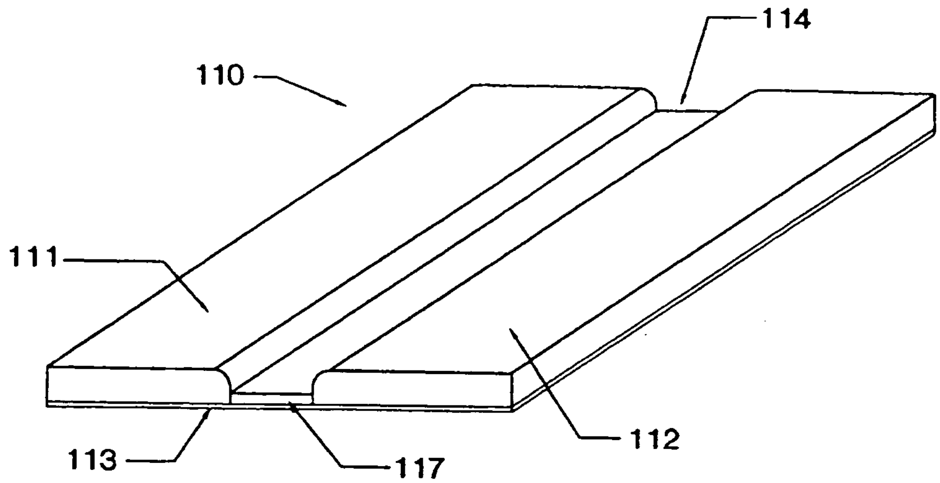


FIG. 9

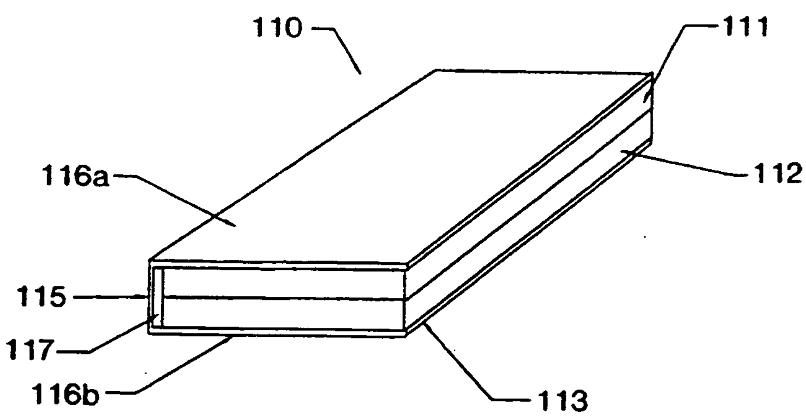
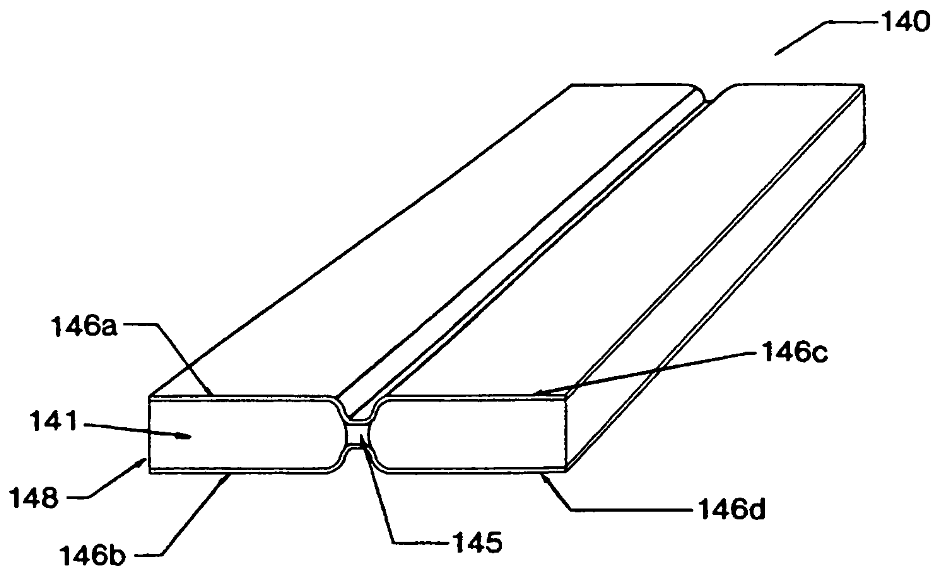


FIG. 10


140
146a
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148
146b
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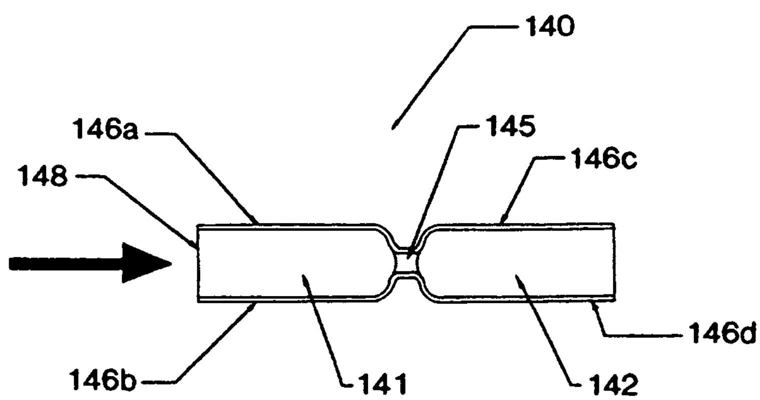
146c
146d

FIG. 11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146a
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146c
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146b
141
142

FIG. 12

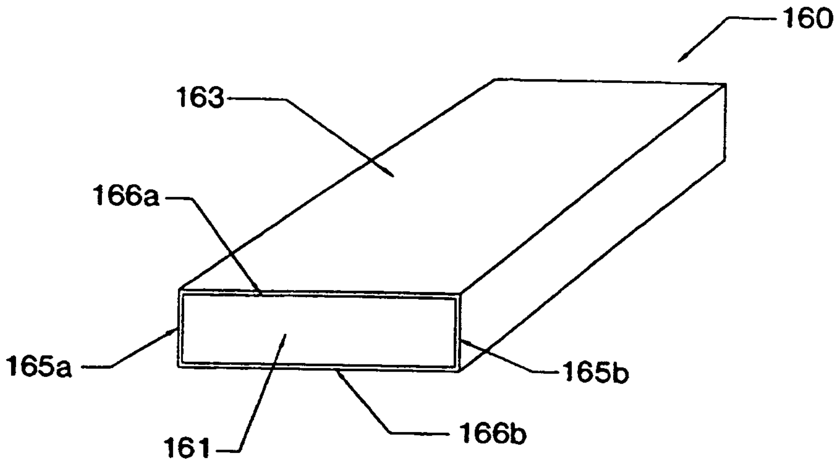

FIG. 13

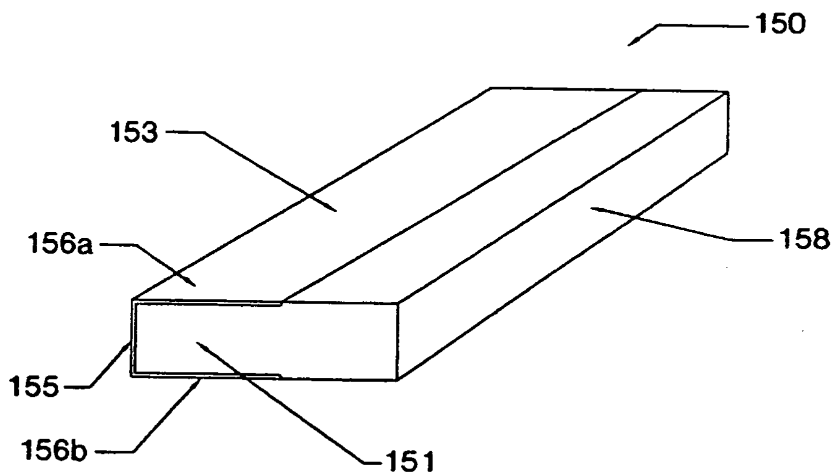

FIG. 14

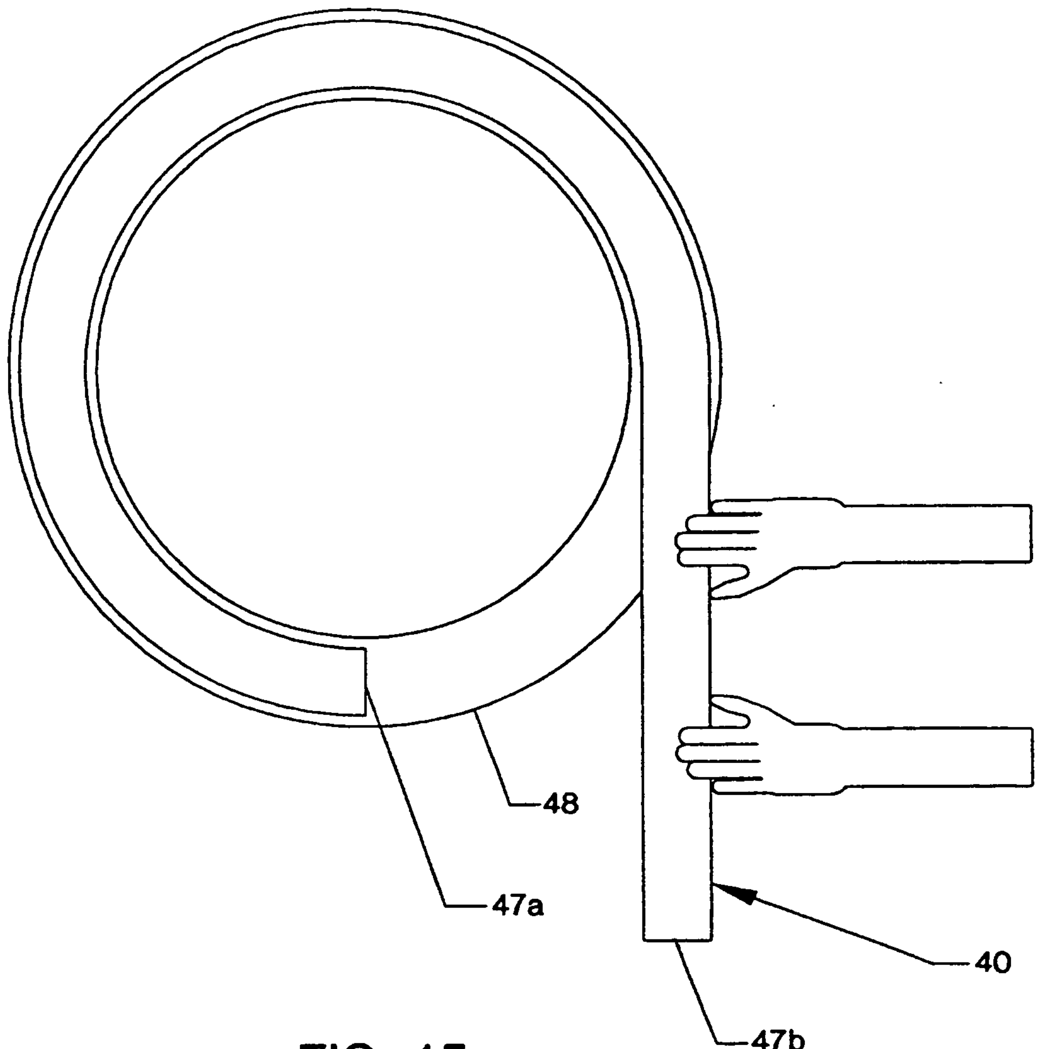

FIG. 15

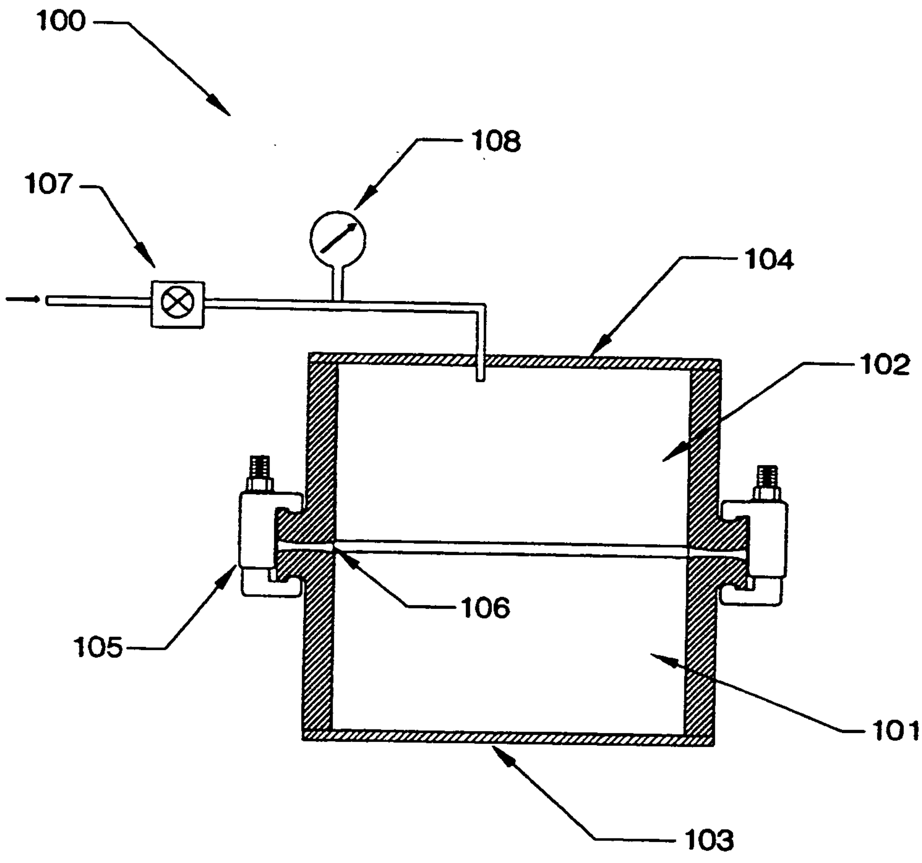

FIG. 16

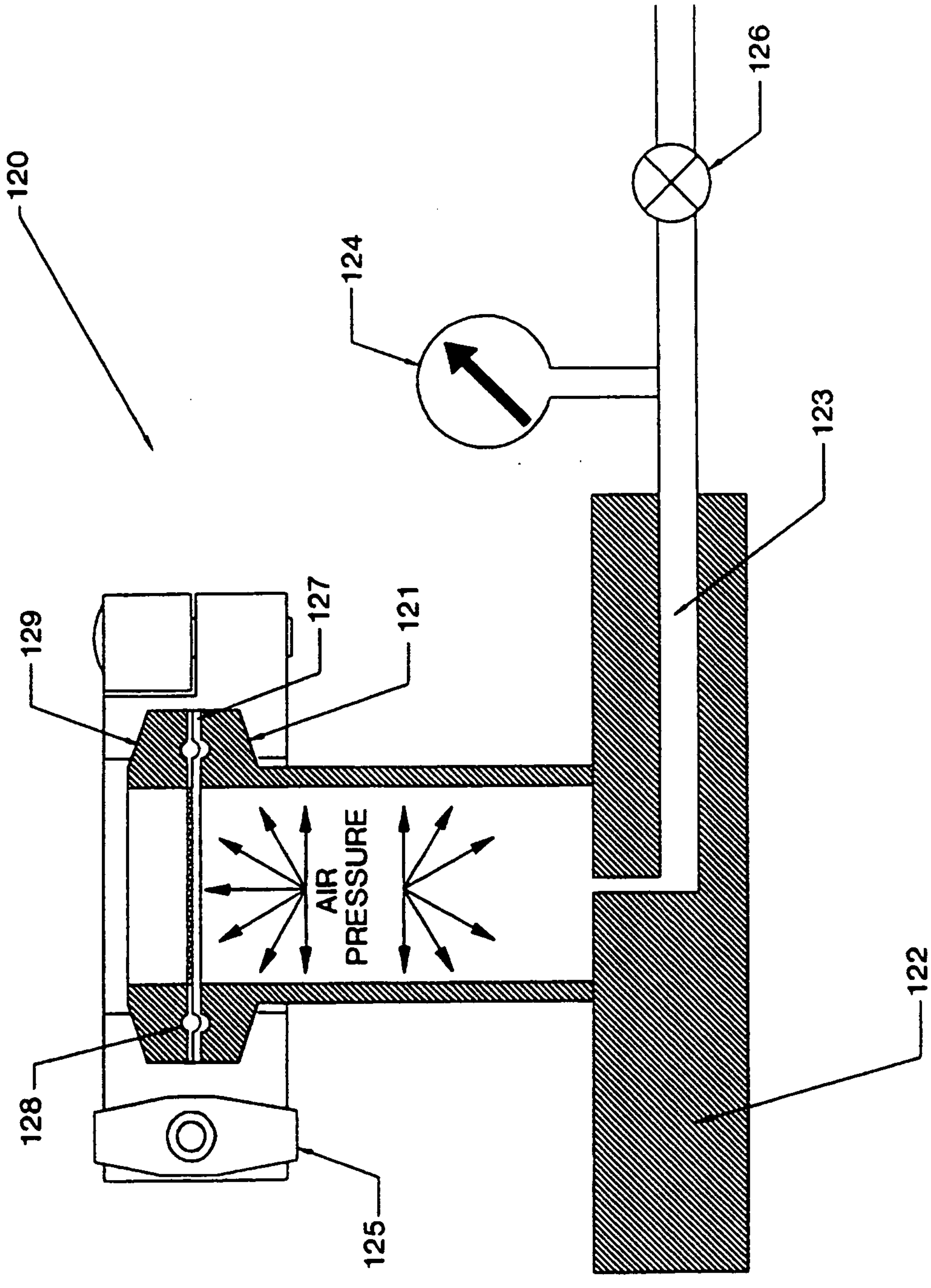

FIG. 17

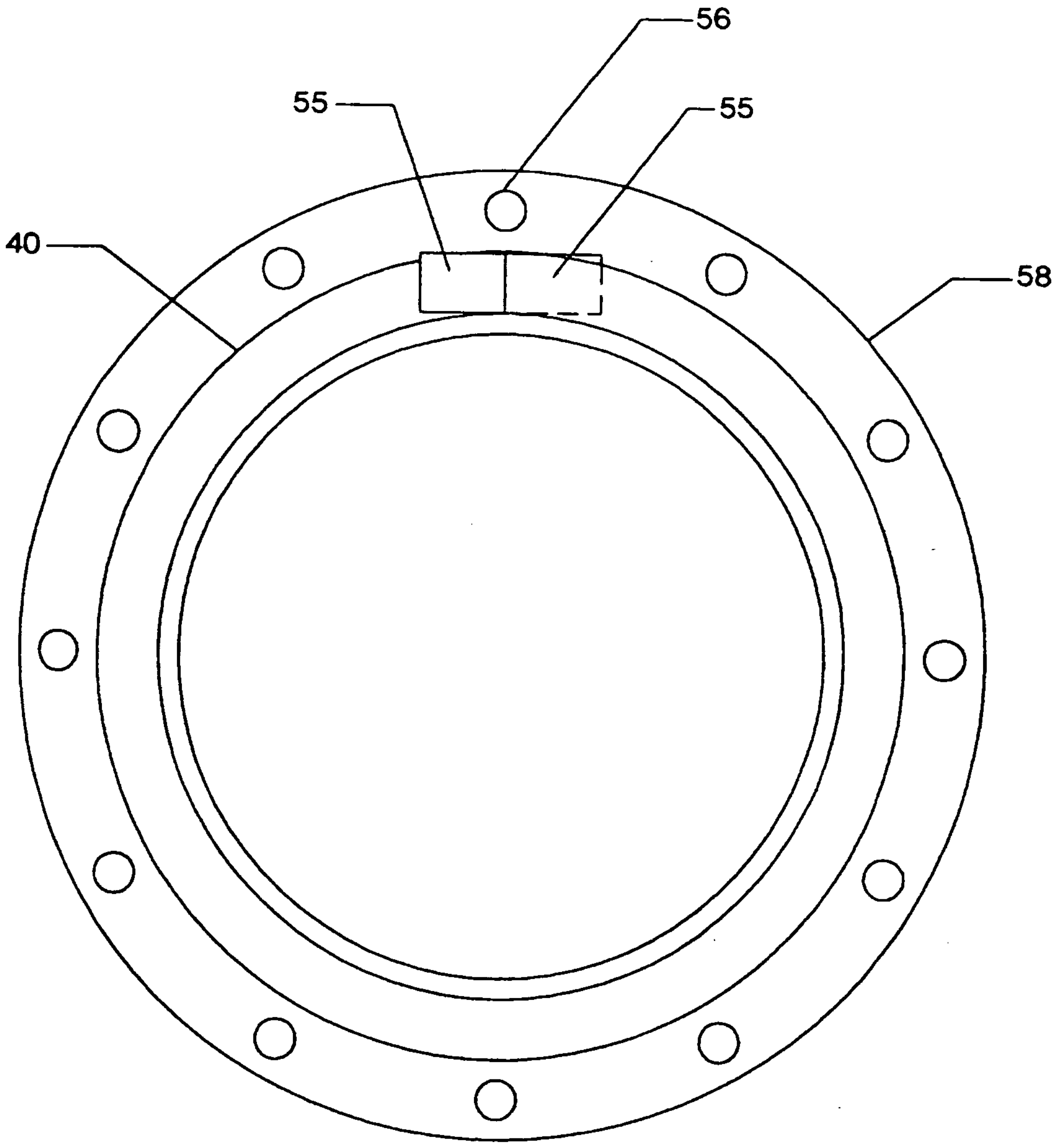

FIG. 18

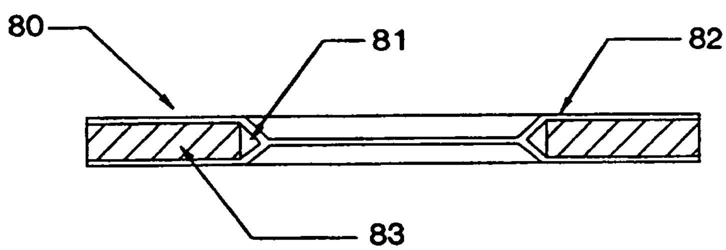

FIG. 19

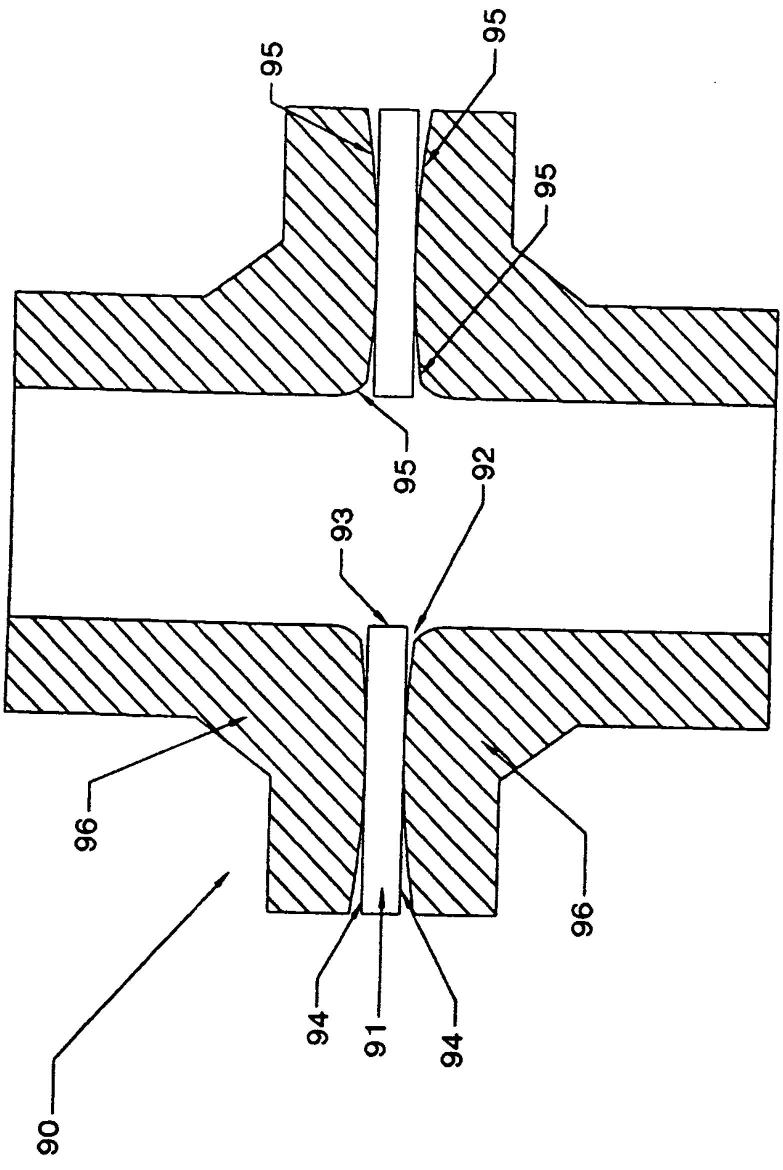

FIG. 20

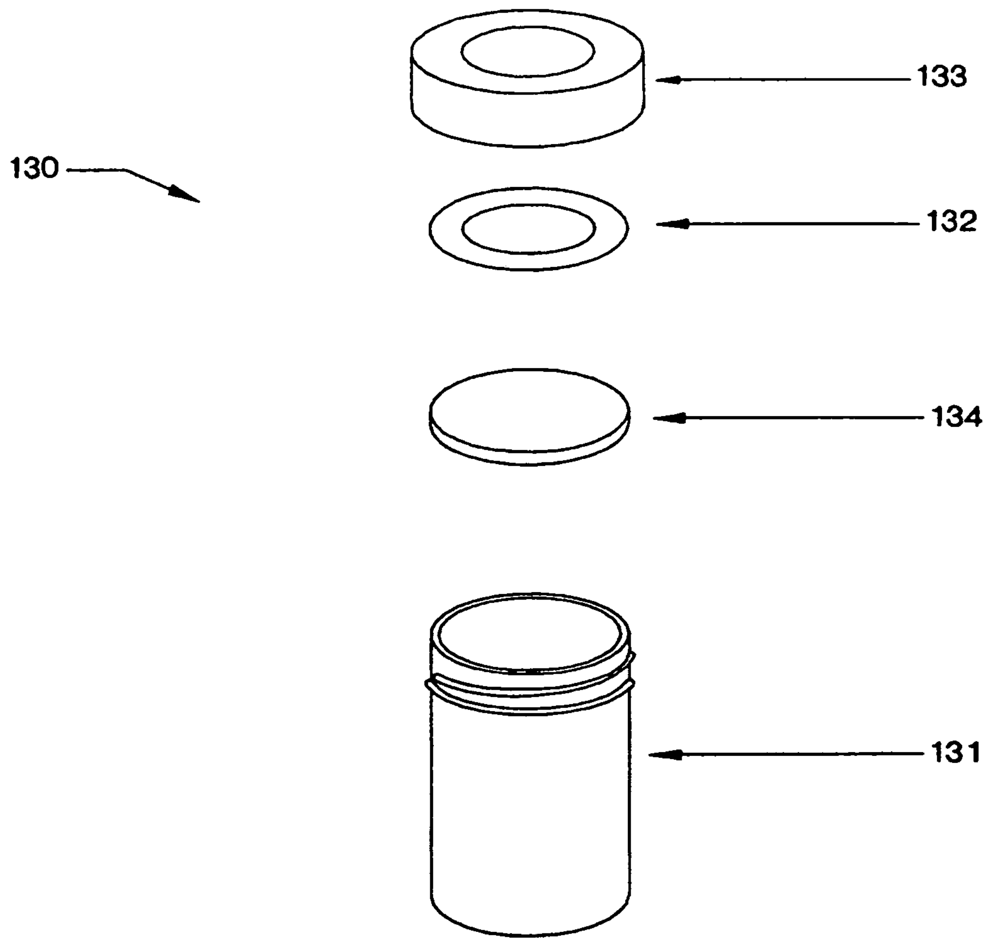

FIG. 21

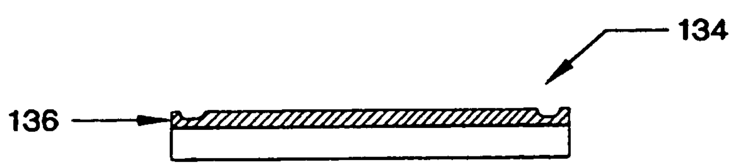

FIG. 21A

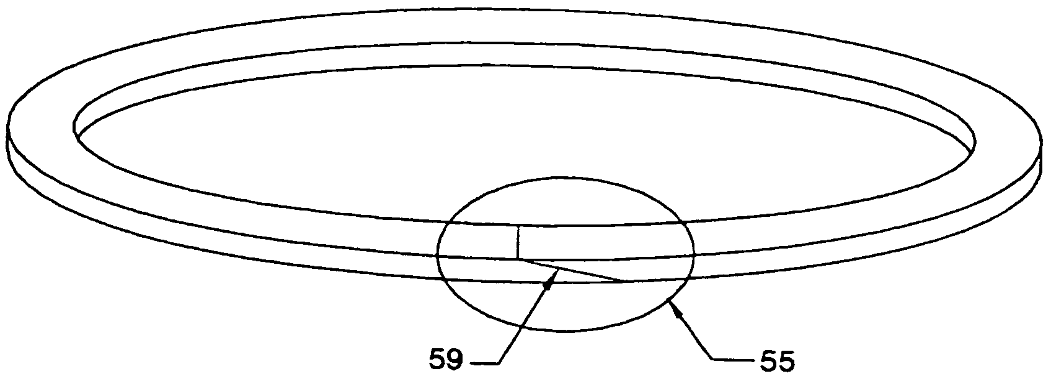

FIG. 22

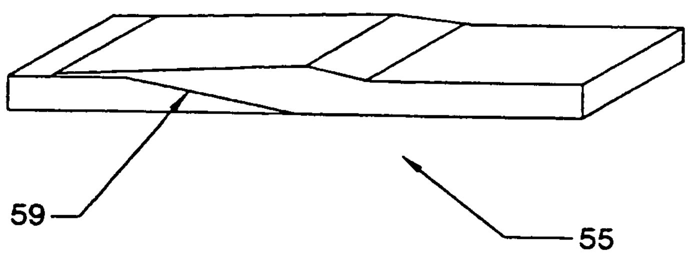

FIG. 22A

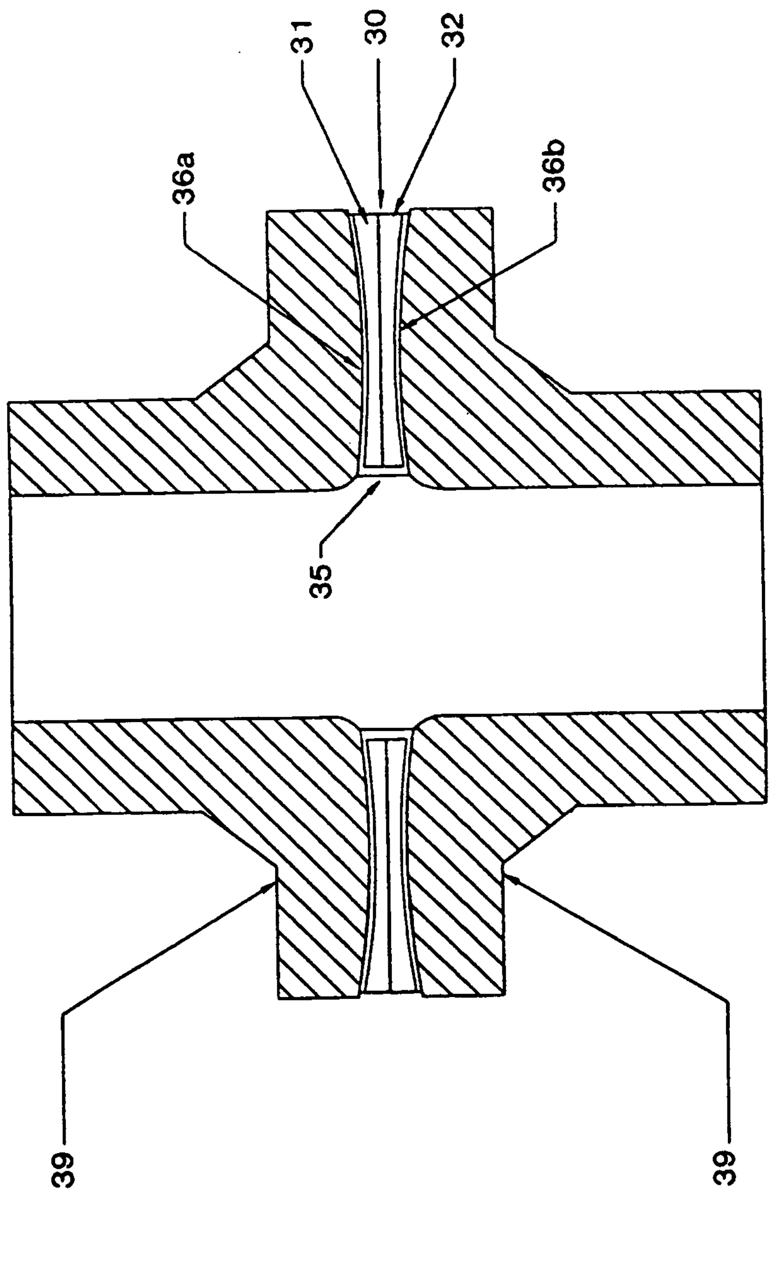

FIG. 23

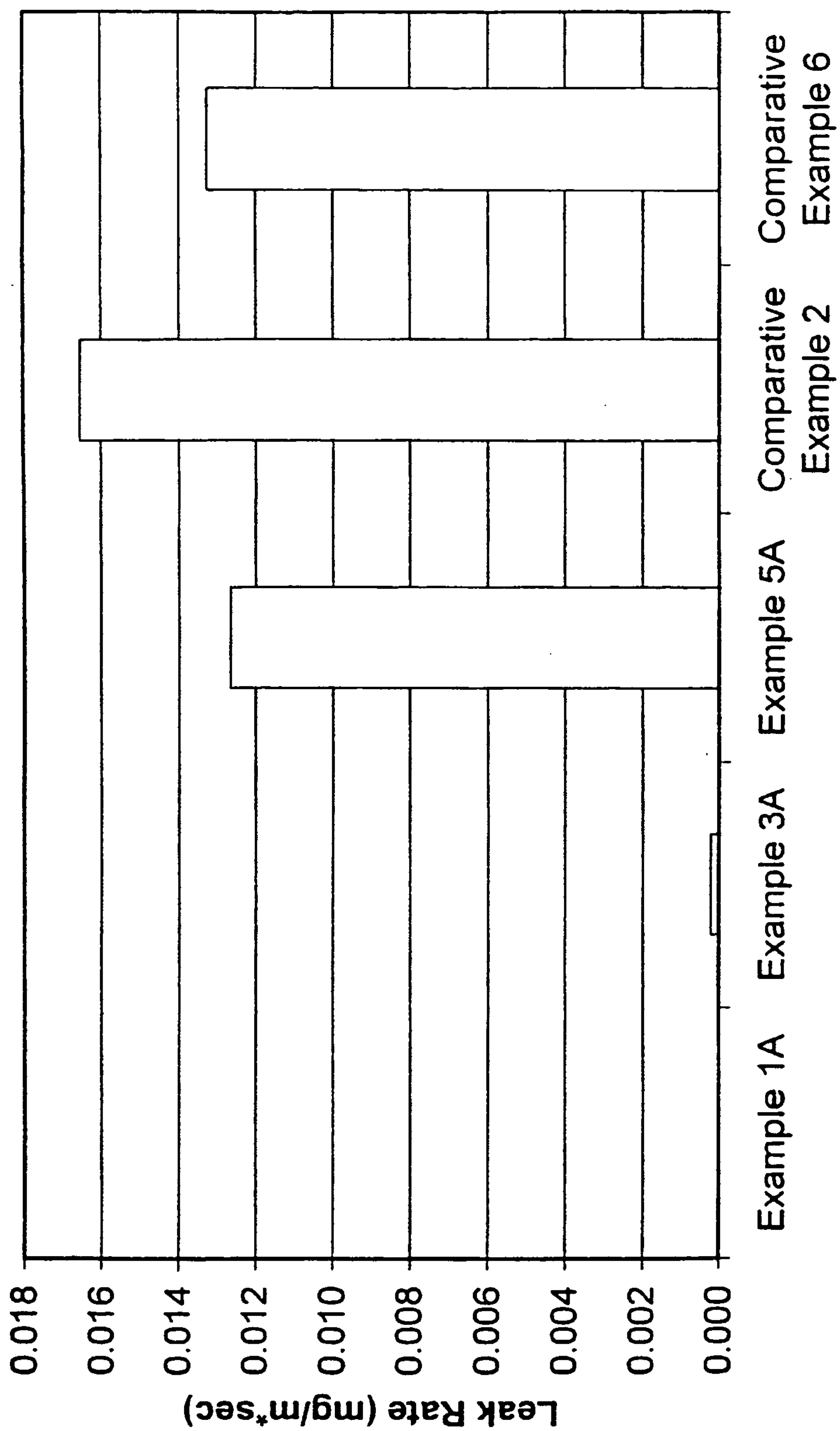
Leak Rate (mg/m*sec)
0.018
0.016
0.014
0.012
0.010
0.008
0.006
0.004
0.002
0.000
Example 1A
Example 3A
Example 5A
Comparative Example 2
Comparative Example 6

FIG. 24

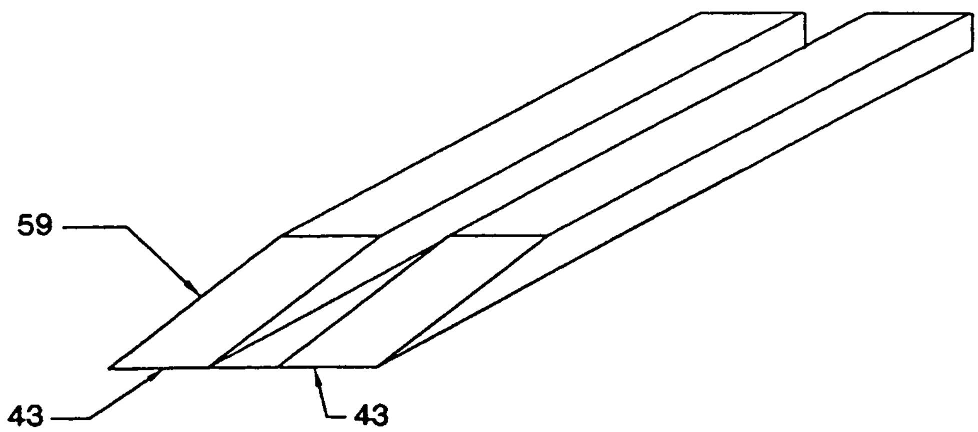

FIG. 25

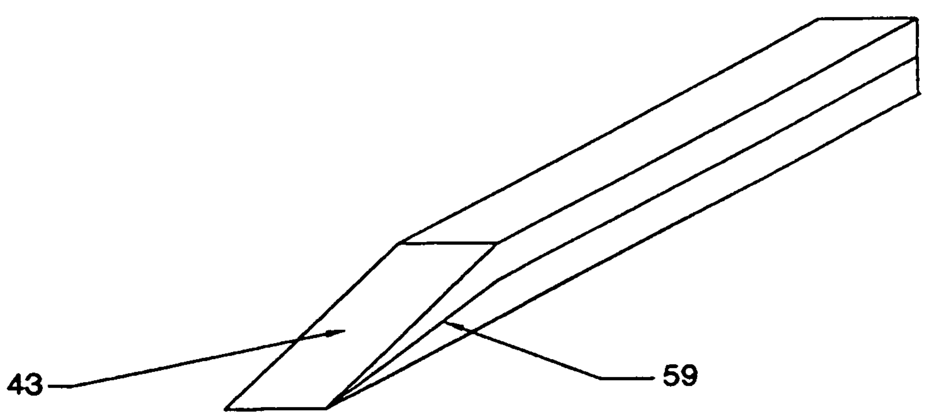

FIG. 26

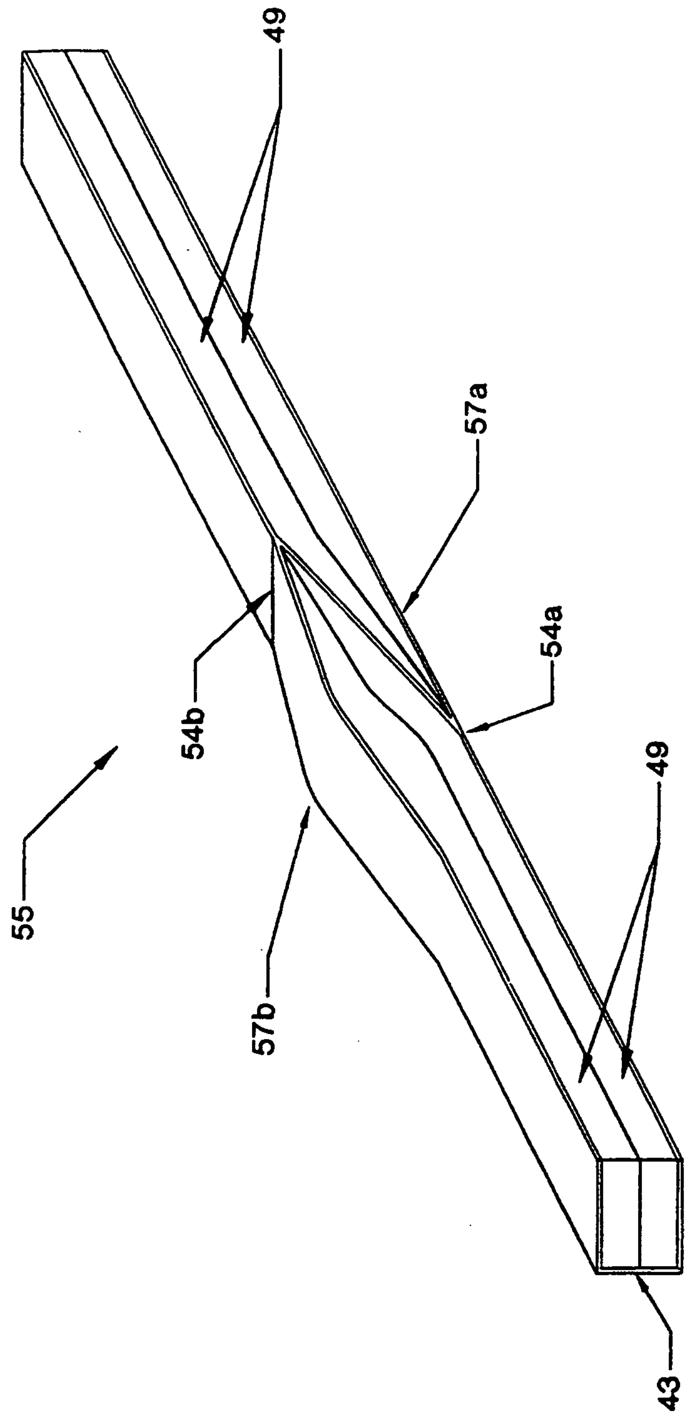

FIG. 27

INTERNATIONAL SEARCH REPORT

Inter al Application No
PCT/US 00/27784

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 F16J15/10

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 F16J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 822 357 A (GORE W L & ASS GMBH) 4 February 1998 (1998-02-04) column 2, line 39-45 column 3, line 15-24 figure 2	1,2,14
A		18
X	US 5 160 773 A (SASSA ROBERT L) 3 November 1992 (1992-11-03) column 1, line 23-26 column 2, line 41-55 figures 2,4	1,14,15
Y		2,3,6,12
A		16

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search: 12 January 2001

Date of mailing of the international search report: 19/01/2001

Name and mailing address of the ISA
European Patent Office, P.B. 5818 Patentlaan 2
NL – 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer
Van Wel, O

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Inter ·al Application No
PC1/US 00/27784

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
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X	US 5 492 336 A (LUBIN CINDY B ET AL) 20 February 1996 (1996-02-20) column 5, line 7-13 column 5, line 51-62 column 8, line 12-39 claims 5,9	1,15
A		7-11
A	PATENT ABSTRACTS OF JAPAN vol. 017, no. 172 (M-1392), 2 April 1993 (1993-04-02) & JP 04 331876 A (NIPPON PILLAR PACKING CO LTD), 19 November 1992 (1992-11-19) cited in the application abstract; figure	1-3,6, 12,14
A	DE 43 22 373 A (CHEMICAL REACTOR SERVICES KEAR) 13 January 1994 (1994-01-13) column 1, line 31-43 figure 1	1-3,16

3

INTERNATIONAL SEARCH REPORT

.ırmation on patent family members

Inter al Application No
PCT/US 00/27784

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			DE	69203605	T	04-01-1996
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